NASA/TM—2002-211507

Thermocouple Calibration and Accuracy in a Materials Testing Laboratory

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April 2002

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Acknowledgments

We greatly appreciate the technical advice and critical review of the manuscript by R. Park of Marlin Manufacturing Corporation. We would also like to thank the diligence of various people who performed the calibration experiments and collected the data: Larry Huse, Eva Horvat, Bill Karpinski, and Sharon Thomas.

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Introduction

Temperature measurement is a critical element in numerous experimental programs for aerospace propulsion and power applications. Typically, thermocouples (TCs) are used for temperature measurements and it is important to ensure that the TCs are accurate and give reliable readings. In the Structures and Materials Divisions, TCs are used for many processes such as furnace control for heat treating, material processing and mechanical and component testing. Most of these processes have stringent requirements on temperature accuracy and stability, as well as on thermal gradients within the test object. These requirements are usually defined in various standards such as those given by ASTM. The purpose of this report is to develop and document TC calibration methods that are suitable for most of the Divisions' needs. The literature [1–3] provides recommended practices and expected results on reproducibility and uncertainties for given methods of calibration. However, there were three shortcomings in these references. The descriptions of the experimental setups are not sufficient for easy replication in the lab. Secondly, some of the steps recommended would be very time consuming. Finally, the statistical analyses to determine the quoted uncertainties in measurements were not provided. Therefore, the documentation of the accuracy associated with specific steps in the calibration process will enable decisions on whether the improved accuracy of a given step is worth the cost. This report also provides a complete statistically-rigorous documentation of the experimental results including the uncertainties assigned to subsequent calibration measurements.

This paper documents the procedure recommended for calibrating longer TCs and TCs made from spools of wire to accuracies similar to those reported in refs. 1–3. It treats the most commonly used TCs, types R and K, in the two Divisions. The bulk of this work was performed with type R (Pt/Pt-13%Rh) TCs. Other types can be calibrated using these procedures as long as the equipment is compatible with those specific types. Also, these processes were conducted in air so TCs containing easily oxidized metals (e.g., tungsten) cannot be used in these processes.

Both sheathed and unsheathed TCs were tested. Sheathed TCs are constructed by placing the TC wire, including the beaded hot junction, within an enclosed tube of either ceramic or metal. This tube protects the TC from handling damage, which can affect its accuracy

[1,2]. Likewise, the sheath reduces environmental contamination that can degrade TC performance. However, due to the insulative properties of particularly the ceramic tube and the finite distance between the TC bead and the point of interest, sheaths may introduce some small temperature errors. In addition, sheathed TCs have a slower thermal response than an unsheathed TC.

Additionally this paper presents in Appendix A, a history of previously attempted procedures, together with their shortcomings. A description of the statistical methods used in analyzing the data is presented in Appendices B-D. It is recognized that much of the content of this report is probably a duplication of numerous investigations over the last 50 years. Nevertheless, we had difficulty finding the data we needed to make recommendations and decided that documentation of all of these experiments in a single report would provide some value.

Experimental Procedure

The basic procedure described in the article uses the comparison method of calibrating TCs, in which unknown TCs are compared to a known, secondary standard. In this case, the hot junctions are placed in close proximity to one another to ensure similar temperatures. The TC used as the secondary standard has been previously calibrated by the manufacturer and is traceable to NIST.

Experiments were conducted in a Lindberg tube furnace (model 54433) capable of temperatures up to 1500 °C. The 3 in. diameter by 36 in. long alumina tube had a wall thickness of 0.13 in. A temperature controller (Eurotherm #818) with 1 °C resolution was used to control the furnace. The controller was set to the adaptive tuning mode. The furnace temperature was controlled using a type R TC inserted into the radial center of the alumina tube from the rear of the furnace. Its hot junction was placed at the midpoint of the furnace length. It was found that furnace control using a TC in the sidewall of the furnace and outside the alumina tube was unsuitable for accurate and stable temperature control of the hot zone. Care is also required in insulating the ends of the furnace tube consistently from run to run.

The TCs used the appropriate type TC mini-connectors. Four feet of TC extension wire was used between the TC and the data acquisition system. The data acquisition system was a Fluke HydraSeries II, which contained an electronic reference point junction. The data acquisition system was calibrated by the NASA-GRC Calibration Lab. It was also spot-checked against a calibrated digital voltmeter that had 1 μV resolution. Data from various TCs were periodically collected and then routed either to a printer or a personal computer for a permanent copy. The setup is shown in Figure 1. Conventional methods were employed to minimize electromagnetic interference (EMI) from the furnace. However at the higher temperatures, EMI still caused some fluctuations (a few tenths of a degree at 1100 °C) in the digital temperature readings.

In order to provide a more stable measurement, the concept of "macropoints" was adopted. A macropoint consisted of the average of approximately10 readings taken at specific time intervals (typically 5 seconds). Table I shows the results of experiments intended to isolate sources of variability in the TC readings. Data are reported as the standard deviation (std. dev.) in the difference between two sheathed-type R TCs from the same batch, in two positions within a metal block. In each run, a set of three macropoints was recorded at 2-minute intervals. Three additional runs were made where the TCs were not moved between the heating/cooling cycles. Four runs were made where the TCs were completely removed and re-inserted after the heating/cooling cycle. Inspection of the individual data points and the summary in Table I resulted in two main conclusions. First, the number of readings within a macropoint need not be as high as 10: reduction to 5 is possible and was practiced in this investigation because of the need to manually transfer data in earlier runs from a printed hardcopy into a data analysis package. More recently, a change in practice to using a computer to directly record the data eliminates the burden of data transference and reduces the need for a macropoint. However, at the higher temperatures (1000 °C and higher) where the effects of electromagnetic interference are more noticeable, a 10-reading macropoint is still recommended. Secondly, the variations (i.e., standard deviations) between macropoints and between runs where the TCs are not physically disturbed are very small. Hence, only a single macropoint is needed to characterize a temperature in a given run. Comparing the standard deviations in Table I clearly shows that a major source for the increase in standard deviation from less than ~0.1 °C to ~0.5 °C or more can be traced to slight differences in the physical positions of the TCs each time they are inserted within the metal block and within the furnace. In subsequent sections, the standard deviations and confidence intervals will be reported where repeats are always defined as complete removal and re-insertion of the TCs between runs.

Table I
Sources of Variability in Sheathed TC Readings
Data reported as the difference between two sheathed type R TCs from the same batch, in two positions within a metal block.

Nominal Temperature (°C)	Std. Dev. for the 10 readings within the macropoint. (pooled over 12 macropoints per Temp.)	Std. Dev. among the 3 macropoints. (pooled, 4 sets of macros per Temp.)	St. Dev. among 3 runs without moving TCs between cycles	St. Dev. among 4 runs where TCs were removed and re-inserted between cycles.
700	0.06	0.06	0.02	0.56
900	0.08	0.03	0.03	0.33
1100	0.13	0.03	0.01	0.33

Fluctuations in the cold junction temperature are mentioned as a major source for error, and ice-baths are frequently recommended [1–3]. The Fluke data logger does have electronic room temperature compensation, which should alleviate this problem. The compensation for differences between the thermocouple wire ends and the data logger input is accomplished by several elements of the circuit, including the connectors, extension wire, and input circuit to the data logger. The effectiveness of those elements was investigated through selective heating of the various components by means of a heat gun. The temperature of the components changed from 23 °C to 40 °C and the change in the TC reading at 700 °C was recorded. The TC connector plugs, the extension wires, and the input card for the data acquisition system were each heated independently. There was a maximum of 0.3 °C change in the displayed temperature, which occurred only when the input card was heated. The changes were smaller when the other components were warmed. Since typical fluctuations in the ambient temperature are significantly smaller than those caused by the heat gun, this setup can be considered stable.

Results and Discussion

A. Sheathed Thermocouples

The final and accepted setup for calibrating sheathed TCs is shown in Figure 2, and is known as a solid block comparison method [2]. To minimize differences in the temperature of the TC junctions, a solid metal block with holes drilled for individual TCs is employed. A 2 in. diameter by 3 in. long cylinder of Haynes 188 (Co-22Cr-23Ni-14W) was chosen for the block material due to its excellent oxidation resistance up to 1100 °C [4]. Seven 9/32 in. x 1.5 in. deep diameter holes were bored into the block. They were located at the 12, 2, 4, 6, 8 and 10 o'clock positions and the 7th TC was placed in the center of the block. For calibrating unknown TCs with this setup, the unknowns were placed in the outer holes and the standard TC was placed in the center position. Care must be taken to be sure that all TCs are bottomed-out. The holes were numbered as shown in Figure 2 and will henceforth be called positions. The block was placed on two ceramic boats that elevated the block such that it was approximately centered within the 3 in. diameter furnace tube (Fig. 2). The block and boats were pushed into the tube until they were centered with respect to the furnace length.

Before TCs could be calibrated, potential errors due to positional bias within the block had to be determined. ("Bias" is the statistical term defined as the offset value attributed to a specific factor.) The positional bias can be added to or subtracted from any difference between a TC in the block and the secondary standard TC to yield the true thermocouple bias (= calibration offset). To determine the positional bias, seven secondary standard TCs which were sheathed in 18 in. long alumina tubes were used. These TCs were made from 20 gauge, type R wire taken from the same wire lot. A lot calibration with NIST traceability was performed by the TC manufacturer [5]. The manufacturer claims that variability within a lot is exceedingly small, which was confirmed in our lab. As described in Section B.3, four TCs from the same lot were welded together to form a common junction, and the temperature difference among the four was only 0.06 °C. Thus all of the TCs from the same lot were treated as identical, and any difference between the positional and the center readings was assigned as positional bias.

To determine the mean positional biases and their uncertainties, the following runs were made. With a TC in each position in the block, the furnace temperature was increased to 700 °C. After thermal equilibrium was reached (approximately ½ hour), data were collected. Thereafter, the furnace temperature was increased to 900 °C and the process repeated. After taking final readings at 1100 °C, the furnace was cooled to ambient. The TCs, block and boats were removed from the furnace. This sequence constituted one run. The set-up was then re-established and the entire process repeated. A total of six runs (repeats) were made. Statistical analyses were conducted on the resultant data. Both a table of the raw experimental data and a full explanation of the statistical analyses conducted are presented in Appendix B. Table II below provides both the calculated mean positional biases as well as the standard deviations of the 6 runs sorted by both position and temperature. These data represent the difference between the positional temperature and the center. Note that the positional temperature at each of the outer positions is hotter than the center of the Haynes block (hence a positive number). The average and standard deviation of these runs are listed in Table III. Note that the positional bias is less than 2 °C in all cases.

Table II
Mean positional biases and their standard deviation for 6 runs.

Position	Mean Positional Bias (°C)		
	700 °C	900 °C	1100 °C
2	1.18	1.36	1.48
3	1.46	1.49	1.62
4	0.92	0.72	0.73
5	0.75	0.57	0.81
6	0.94	0.73	0.81
7	1.26	1.33	1.45

Position	Standard Deviation (°C)		
	700 °C	900 °C	1100 °C
2	.41	.36	.37
3	.40	.30	.26
4	.50	.41	.33
5	.47	.40	.26
6	.44	.44	.38
7	.51	.42	.36

The statistical analyses found that for each temperature, the difference in the top positions (4,5,6) was statistically insignificant. Likewise, the difference in the bottom three positions (2,3,7) was statistically insignificant. However, the top three positions were significantly cooler than the bottom three positions. Finally, a given position showed no significant differences as a function of temperature, so the position bias values for the three temperatures could be pooled into a single number covering the entire temperature range.

These results prompted us to divide the mean temperature bias of the positions with respect to the center temperature into two groups: top and bottom. The standard deviations in Table II above were used to construct 95% Confidence Intervals (CI) on these mean positional biases. The details are presented in Appendix B and are simply summarized in Table III. The resultant positional bias will be subtracted from the actual TC readings for future, unknown TC calibrations when they are compared to a known, standard TC located in the center of the block.

Table III
Position Bias Values and 95% Confidence Intervals (in °C) for Sheathed TC Calibration.

Top positions:	0.78 ± 0.21
Bottom positions:	1.40 ± 0.21

In summary, the procedure for calibrating an unknown, sheathed thermocouple is as follows. First, a known TC with NIST traceability is positioned in the center of the comparator block, and the unknown TCs are placed in positions around the circumference. It should be noted that the TC used as the standard will come with its own calibration constant, which must be taken into account to calculate the true temperature.[1] After stabilizing at a given temperature, a macropoint is taken and then ΔT= Unknown TC-Standard TC is calculated. The position bias is then read from Table III and subtracted from ΔT. Lastly, the calibration adjustment for the standard TC is then subtracted, thus providing a calibration constant for the unknown TC. The 95% CI on this calibration constant is ± 0.91, as described in Appendix B. If the calibration experiment is repeated (including removal and re-insertion of the TCs into the furnace), and the results of the two experiments are averaged, the 95% CI on this value is ± 0.66.

It is believed that slight shifts in the position of the TCs inside the metal block are the main sources for error. A shift in the longitudinal thermal gradient from the beginning to the end of the testing trials is another potential source for error. In the course of running the numerous experiments in this effort, a few isolated examples were found where it appeared that the scatter in the results from two repeat TC calibration runs was significantly greater than the confidence intervals quoted in the previous paragraph. Inspection of the data showed that the TCs in the 6 surrounding positions agreed with each other in both repeat runs, but their agreement with the center TC was significantly different between the two runs. This situation is almost certainly due to the depth of the

[1]This analysis assumes that we know the calibration constant of the standard TC perfectly: the standard deviation of this number is given as zero. The manufacturer does not provide calibration data with comparable statistics for use in this analysis.

center TC within the metal block having shifted more than usual in one of the runs. Thus, the center TC, which is the standard against which all the unknowns are compared, is equally likely to suffer from errors due to slight differences in how the TC is inserted within the metal block. Because of this effect, it is recommended that a second standard TC be used in a calibration run, placing it in one of the outside TC positions, to act as a double check of the calibration run. The two standard TCs must agree with each other for the run to be valid.

B. Unsheathed Thermocouples

B.1 Metal Block Method

In principle, the same exact setup used for sheathed TCs can be used for unsheathed TCs. Despite the fact that we have not run any confirmation experiments, we recommend that the data in Table III be used to calculate the calibration constant for an unknown, unsheathed TC if the setup described in Section B is used. However, several additional options were explored for the case of unsheathed TCs. The first and most important is that a different tube furnace was used in order to match the most commonly used unsheathed TCs in the Materials Division mechanical testing labs. In these labs, shorter, 15 in. long type R TCs are frequently used in conjunction with 16 in. long, 2.75 in. internal diameter furnaces. In order to minimize the extent of immersion error in the determination of a calibration shift, a smaller tube furnace that matched the mechanical testing furnaces was used. Others [6] have shown that with type R TCs after extended use during creep testing, apparent calibration shifts can be mostly attributed to immersion effects, rather than a true shift in the TC wire.

Since this particular tube furnace was equipped with a 1.25 in. diameter quartz tube, a 1 in. diameter metal block with smaller 1/8 in. diameter holes was used to better match the dimensions of both the furnace and the unsheathed TCs. The positions for all experiments with this block were slightly different than for the sheathed block, as shown in Fig. 2. In order to maximize furnace life, all experiments were limited to 1000 °C. To produce more data in a given run, experiments were started at 600 °C and incremented in 100 °C steps. In all other details, the data recording protocol using macropoints was identical to that used previously.

The statistical analysis for these experiments is contained in Appendix C. It was found that the behavior of this new furnace/TC combination showed subtle differences compared to the sheathed experiments. First, the position bias of the TC holes was not a simple vertical gradient through the cross section of the metal block, as it was for the sheathed experiments. Rather, the position bias showed a slanted gradient with the two upper right positions being slightly cooler than the two lower left positions. This gradient is apparently the result of the clamshell furnace design. Second, while the mean values for the position bias were still independent of temperature, the standard deviations attached to these positions increased as temperature increased, as shown in Table CIII of Appendix C. Recall that the experiments on the sheathed TCs exhibited standard deviations that were constant over the temperature range.

In summary, the procedure for calibrating an unknown, unsheathed thermocouple is as follows. A known TC with NIST traceability is positioned in the center of the comparator block, and the unknown TCs are placed in positions around the circumference. It should be noted that the TC used as the standard will come with its own calibration constant, which must be taken into account to calculate the true temperature. As mentioned in Section A, a second standard TC placed in one of the circumferential positions adds another degree of confidence in data interpretation. After stabilizing at a given temperature, a macropoint is taken and then ΔT= Unknown TC-Standard TC is calculated. The position bias is then read from Table CIV in Appendix C and subtracted from ΔT. Lastly, the calibration adjustment for the standard TC is then subtracted, thus providing a calibration constant for the unknown TC. The 95% CI on this calibration constant is ± 0.56 at 600 °C and ± 1.35 at 1000 °C, as described in Table CV in Appendix C. If the calibration experiment is repeated (including removal and re-insertion of the TCs into the furnace), and the results of the two experiments are averaged, the 95% CI on this calibration constant is ± 0.32 at 600 °C and ± 0.76 at 1000 °C. Note that compared to the CI's measured with the sheathed TCs, this method produced better results at 600 °C but was slightly worse at 1000 °C.

B.2 Tying Method

An alternate method of tying the hot junctions of the unknown TCs to a standard TC was also investigated. This was done to eliminate position bias and still be able to calibrate multiple TCs in one run. Tying was best accomplished by threading a loop of wire (we used 0.012 in. diameter type K TC wire) through one leg of the TCs and twist-tying the TCs moderately tight. A second loop of wire was then wrapped around the hot junction beads to ensure tight contact between the unknown TCs and the standard TCs (see Figure 3). After tying, care was taken to make sure the individual wires of each TC were not touching each other away from the bead. Some degree of skill and practice is required to perform the tying adequately. If the tying is too loose, or the TC wires are touching in undesirable locations, more data scatter occurs. If the tying is too tight, damage to the TC is likely. By tying TCs together an electrical connection can be made at areas other than the bead and this can create a source of error in the measurements. Since the length of exposed wire (i.e., the distance between the bead and the first ceramic insulator) is small (approximately 1/8 in.), it is assumed that the temperature is isothermal over this distance and the error is negligible, but is a consequence of trying to maximize efficiency by calibrating many thermocouples simultaneously.

The smaller furnace and the identical TCs used in Section B.1 were used for the calibration experiments. Because of the concern that operator skill would influence the results, three operators performed five repeat runs (2 runs each by 2 operators, and 1 run by the third), and the results were combined. The complete statistical analysis is presented in Appendix D. With the tying method, there is no Position Bias that needs to be calculated, thus making the results easier to analyze. As was the case for the metal block experiments described in Section B.1, the standard deviations characterizing the scatter in these experiments increased as the nominal temperature increased. In summary, the procedure for calibrating an unknown, unsheathed thermocouple is as follows. First, a known TC with NIST traceability is tied to as many as three unknown TCs. It should be noted that the TC used as the standard will come with its own calibration constant, which must be taken into account to calculate the true temperature. After stabilizing at a given temperature, a macropoint is taken and then ΔT= Unknown TC-Standard TC is calculated. The calibration adjustment for the standard TC is then subtracted, thus providing a calibration constant for the unknown TC. The 95% CI on this calibration constant is ± 0.56 at 600 °C and ±1.11 at 1000 °C, as described in Table DIV in Appendix D. If the calibration experiment is repeated (including removal, untying, and re-tying the TCs), and the two experiments are averaged, the 95% CI on this value is ± 0.39 at 600 °C and ± 0.77 at 1000 °C. Note that compared to the CI's measured with the sheathed TCs, this method produced a better CI at 600 °C but a slightly worse CI at 1000 °C. The results are virtually identical to the metal block experiments with the same small furnace described in Section B.1. Again, it is believed that slight differences in the tying of the TCs are the main source for error. A shift in the longitudinal thermal gradient from the beginning to the end of the testing trials is another potential source for error.

B.3 Welding Method

References 1 to 3 also discuss a method of welding the unknown TCs to the standard TC in order to enhance the thermal homogeneity between the TCs. Such intimate contact would eliminate the errors associated with shifting positions within a metal block or the reproducibility of tying the TCs together. There is a common misperception that the act of welding a hot junction bead, cutting the bead off, and re-welding the bead can affect the calibration constant of the TC. This concern is counter to the recommendations in refs. [1–3] and private communications with representatives from NIST and ASTM. However, the disadvantage remains that continued welding and cutting beads will serve to shorten the available wire. For this reason, our efforts in investigating this method were limited to a single trial of welding 4 TCs from a single batch. The four TCs exhibited extremely good agreement with each other, producing a standard deviation of only 0.06 °C at all temperatures between 600 and 1000 °C. This value is smaller than the standard deviation obtained by tying the same 4 TCs by about a factor of 3. Assuming that this ratio would be maintained if a full series of repeat experiments were conducted, a CI of approximately ± 0.2 °C for a single calibration run and approximately ± 0.1 °C for a duplicate run would be expected. These results represent the best accuracy and precision obtained in this laboratory. In fact, at this level of error, we may be testing the variability of some other part of our experimental setup, such as electromagnetic interference, the recording devices or the extension wires.

C. Summary and Verification of Type R Thermocouple Calibration Experiments.

A summary of the calibration experiments and the statistical uncertainties (expressed as 95% CI's) expected from these experiments is displayed in Figure 4. The least accurate method of knowing a thermocouple's true value is relying on the manufacturer's tolerance, which is given as ± 3 at 600 °C and as ± 5 at 1000 °C [2]. Figure 5 shows calibration results for 6 separate batches of new TCs purchased over a 2 year period. The range of data covers approximately $\pm$ 1 °C at 600 °C and $\pm$ 2 °C at 1000 °C, which is much tighter than the quoted ranges shown in Figure 4. Thus, the quoted ranges are clearly conservative and the risk of relying on such data appears minimal. However, the benefit of tighter confidence intervals from calibration is seen in Figure 4. Using comparison methods for calibration, ref. [2] quotes CI = $\pm$ 1.2 °C (at 600 °C) as the uncertainty to be expected using good practice, whereas ASTM E220 quotes CI = $\pm$ 0.6 °C at the same temperature [3]. R. Park, a member of the ASTM committee responsible for TC calibrations, recommended [7] that the CI = $\pm$ 1.2 °C value quoted in ref. [2] be used, because it is more recent than the current version of ASTM E220, which was written in 1986. He also mentioned that more statistically rigorous values for uncertainties will be available in a new revision of ASTM E220, which is due to be released in the near future. Further improvements in accuracy and precision can be obtained by calibrating against the melting point of pure metals [2]. Also shown in Figure 4 are the CI's that resulted from the experiments described in Sections A and B. These values are comparable to what is expected from the literature.

The final set of bars in Figure 4 describes another set of experiments where new unsheathed TCs were tested in identical experiments as a "verification run" to see if the standard deviations in practice were consistent with the initial "baseline" runs. First, two batches of 3 TCs each were calibrated in 5 repeat experiments, producing a CI = $\pm$ 1.47 °C at 600C °C and $\pm$ 2.81 °C at 1000 °C. These values were greater than the baseline data described in Section B. This change may be due to either a change in operators or a shift in the longitudinal temperature gradient in the tube furnaces, since it was found after the experiments were completed that both furnaces had heating elements that had degraded. More positively, one of the TC batches was independently calibrated by the manufacturer [5], and the results from the two labs agreed within experimental scatter. Another batch of new TCs were calibrated by the tying method, and a batch of used TCs from the Materials Division Creep Lab were calibrated via the metal block method. Both of these batches had similar CI results as the first batch. The results of these "verification" runs are somewhat equivocal. The larger CI values of the verification runs compared to the baseline runs were clearly undesirable, but the differences were borderline in terms of statistical significance. Furthermore, the agreement with the external lab, and the still relatively small values for scatter, would seem to make the calibration testing worth the effort, certainly compared to the manufacturer's tolerance.

To check on the stability of the metal block calibration method and the position biases over time, the tube furnace used for the unsheathed TC experiments was repaired and after a twelve month waiting period, a new set of calibrations were run. First, seven new TCs from a single batch were used to recalculate the position biases using the small metal block. The standard deviation among these 4 runs was equivalent to the initial baseline runs, thus confirming that accuracy of better than ± 1 °C is achievable. However, the position biases showed considerable shifts, of up to 2 °C from the baseline values in two of the six positions, compared to the other four where the position bias remained within approximately ± 0.5 °C. At first glance, these shifts in position bias could be attributed to the fact that new heating elements were installed during the furnace repair. However, close inspection of the individual data on specific TCs lead us to the conclusion that the difference in position bias between the new runs and the baseline were due to experimental scatter rather than a true change in thermal gradient. If a TC was placed in one of the four 'more stable' positions, its calibration constant was within ± 1 °C of that determined 12 months earlier, and also close to the data supplied by the manufacturer. However, if the TC was placed in one of the two 'unstable' positions, the calibration constant differed from the prior data by ± 2 °C (i.e., subtracting the position bias did not produce consistent results with the baseline).

Therefore, it appears that there is still a source of variability that occurs sporadically and causes data to occasionally fall outside the statistical confidence limits defined in sections A and B and Figure 4. Thus, the presumed benefit of improved accuracy from specific calibration runs may be illusory. Determining the true size of the variability would require several repeat runs that might not be worth the cost. The entire focus of this work was to establish methods for simultaneous calibration of multiple TCs in order to minimize cost. The number of repeat runs necessary to ensure accuracy appears to negate any efficiencies in these multiple TC runs. We therefore conclude that the welding method is the most accurate and cost effective method for calibrating multiple TCs in a single run. If unsheathed TCs are to be used, the tying method with a small number of TCs is likely to be accomplished with the accuracy exhibited in Figure 4, provided that a specific operator has been properly trained and passed qualification standards. For sheathed TCs, the only choice appears to be the metal block comparison method, but achieving the accuracy quoted in Figure 4 appears to require attention to exact detail in experimental setup; additionally, a check for any shift of the position bias values from the baseline should be performed immediately prior to calibrating.

D. Type K Thermocouples.

Type K thermocouples can be used in air (for short periods) up to 1250 °C [2]. For extended periods there is a much lower temperature limit, and this limit also depends on the wire diameter. These thermocouples are subject to errors induced by oxidation or by "short range ordering." Both of these effects cause an accumulative drift in the EMF output of the TC over time. The effect of short range ordering is seen up to 600 °C, and can cause changes up to 3 °C [2]. At temperatures above 1000 °C, the drift due to oxidation can be greater than ~10 °C after a few 100's of hours [8]. The low cost and moderate stability of type K thermocouples usually imply that these thermocouples should be discarded rather than recalibrated. The recommended standard practice is that any portion of the thermocouple that is exposed to elevated temperatures during the test should be removed at the end of the test and a new measurement junction made.

Calibration experiments were performed on TCs made from type K wire (0.012 in. diameter) taken from a spool in the Structures Division Fatigue Lab and calibrated against a type R TC by the tying method. Calibrations were performed on five TCs and a CI = ± 1.3 at 600 °C was determined. The calibration constant of –1.4 °C is valid for the entire spool, although periodic crosschecks will be performed as the spool is consumed.

Compared to the manufacturer's tolerances of $\pm$ 10 °C [2], the value of performing the calibration on the spool of wire is obvious.

Because stability data on the exact wire diameter used in the mechanical testing labs were not found, individual experiments were necessary to quantify the calibration drift to be expected in our labs. Experiments were performed by tying unsheathed, 0.012 in. wire diameter, type K TCs to a type R standard and exposing them in a tube furnace in air. The data from Dahl [8] in Figure 6 shows that calibration drift is negligible at temperatures below about 600 °C. Our own experiments at 300 and 500 °C confirmed this finding. Very little evidence for calibration drift due to short range ordering was found in this lower temperature range: if the effect was present, its magnitude was less than a degree and its duration was confined to the first few hours of the test. Apparently, the different thermal responses of the TCs, and the difficulty in achieving a stable temperature in this low temperature region masked the effect.

Our data from higher temperature exposures are shown in Figure 7. At all 3 temperatures, there is a transient period where the calibration drift changes by about 2 °C during the first few hours of exposure. After this transient the TC was very stable at 700 °C. The TC continued to drift at 800 °C and 900 °C, reaching a total calibration shift after 1000 hours of 5 °C and 6 °C, respectively. Comparison of our data to that of Dahl's is not straightforward because of different wire diameters and exposure temperatures. The closest match is shown in Fig. 8, where Dahl's data at 871 °C is compared to our data at 800 and 900 °C. Figure 8 shows that both data sets are consistent with each other. There are general trends of larger calibration shifts exhibited by either higher temperatures or thinner wires, as would be expected for an oxidation mechanism. The reproducibility between two runs at 800 °C is also shown to be within 1 °C after 1000 hours.

The above experiments all reflect isothermal conditions. Dahl [8] showed abundant data that the effects of oxidation can cause calibration shifts that are dependent on temperature. For example, aging 0.026 in. diameter wire at 871 °C for 1000 hours caused a calibration shift of 4° at 871 °C. The same 871 °C exposure resulted in calibration shifts of 5° at 300 °C and only 1° at 550 °C.

Several stability experiments were run with metal-sheathed TCs to determine if they provide any advantage by protecting the TC wires from oxidation. Two separate sheathed geometries were examined: 1) 0.05 in. diameter wire sheathed in a 0.25 in. diameter Inconel 600™ tube, where the TC was not grounded to the sheath and was separated from the sheath by MgO insulation; and 2) a 0.005 in. diameter wire sheathed inside a 0.032 in. diameter 304 stainless steel tube, where the hot junction was grounded, indicating intimate contact with the sheath. Figure 9a shows that at 800 °C, the larger sheathed TC did indeed stabilize at a slightly smaller calibration shift, but the effect was not very pronounced. More surprising was the behavior of the smaller sheathed TC, which showed no evidence of stabilization even after 1350 hours. This behavior was confirmed in a second run, Figure 9b, and is caused by diffusion in the fine wire diameter in the grounded TC. At 900 °C, the effect is even more pronounced, and the unsheathed

TC is clearly more stable due to its larger wire diameter than the small-sheathed, grounded design (Fig. 10). Additional experiments were run with ungrounded TCs having 0.040 in. and 0.062 in. diameter sheaths and 0.010 in. diameter wire. They showed calibration shifts of 3 and 4 °C, respectively throughout the 1000 hours exposure at 1100 °C. These calibration shifts were accumulated gradually throughout the exposure with no evidence of stabilization at longer times. Hence, the choice of wire diameter for any type of TC is important.

Comparisons between type R and type K TCs are summarized in Figure 11. The manufacturer's tolerances for type R TCs are clearly superior to type K at both at 700 and 900 °C, so type R would be the choice using this criterion. However, a spool (or batch) calibration brings the uncertainty levels achievable in the two TC types to more comparable levels. The next criterion to consider is thermal stability. It appears from the data in Figures 6–9 that 700 °C is a reliable, conservative upper limit for the use of type K TCs in air. This temperature is dependent on wire diameter, but it is certainly valid for wire diameters at least as small as 0.010 in. If one uses the calibration constant after about 1–2 hours of exposure, the stability of the type K TC is excellent. Although the type R TC is also excellent, it can suffer from calibration shift due to cold work [1–3, 6]. Evidence for the magnitude of this shift was supplied by testing six used TCs from a Materials Division creep lab. The temperature and duration of the exposure given to these TCs is unknown, although it is known that at least one TC was used for 10,000 hours at 980 °C. The calibration shifts measured on these TCs are given by the data points in Figure 11 and ranged up to 4 °C. Unfortunately, it is difficult to separate a true calibration shift from the effects of immersion, as is made strikingly clear by Desvaux [6]. The TC must be cleaned and annealed and then recalibrated in order to see if a chemical composition change in the TC wires was causing the measured drift. Furthermore, it is not clear whether all or parts of a calibration shift due to cold work is actually affecting the temperature measurement *in-situ* in the creep-testing machine. In principle, all the cold work should be located in the ~50 mm flexible portion of the TC, which is exposed in the nearly isothermal section of the creep machine furnace. Our recommendation would be that *in-situ* calibration is probably the easiest way of addressing these concerns. In an in-situ calibration, one fresh TC is placed near the used TCs in the same location in the testing furnace, rather than removing the TCs and placing them in a separate 'calibration' furnace.

This discussion indicates that if the calibration shifts of 1–4 °C are critical to the success of an experiment, recalibration of type R TCs should be carefully considered. A measurement of calibration drift with sufficient accuracy appears to be quite difficult and certainly has hindered our efforts to come up with an inexpensive standardized method. An extensive database provided from a series of *in-situ* calibrations would be needed before a final recommendation can be reached. The type K TCs have an advantage in that they are used only once and the calibration constant from the spool is valid for all tests below about 700 °C or up to ~900 °C for times less than 20 hours, again depending on wire diameter.

Conclusions

This paper has provided a consolidation of information that can be used to define procedures for enhancing and maintaining accuracy in temperature measurements in materials testing laboratories. These studies were restricted to type R and K thermocouples (TCs) tested in air. Thermocouple accuracies, as influenced by calibration methods, thermocouple stability, and manufacturer's tolerances were all quantified in terms of statistical confidence intervals. By calibrating specific TCs the benefits in accuracy can be as great as 6 °C or 5X better compared to relying on manufacturer's tolerances. The results emphasize strict reliance on the defined testing protocol and on the need to establish recalibration frequencies in order to maintain these levels of accuracy. Type K TCs are best utilized when discarded after a single use. A limited number of calibrations on individual TCs are sufficient to characterize the entire spool of TC wire. After some initial transients during the first hour or two of exposure, Type K TCs are stable for thousands of hours below 700 °C but only for about 20 hours at and above 900 °C, though the exact temperatures are highly dependent on wire diameter. Type R TCs are more stable at the higher temperatures, but may need periodic re-calibration as accumulating cold work during use causes calibration shifts. However, recalibration is complicated by immersion errors and it may be more efficient to replace the TCs with new ones. Although an extensive effort was made in using the metal block and tying variants of the comparison method for TC calibration, the welding method clearly exhibited the best reproducibility and accuracy. Finally, a consideration of *in-situ* calibration appears to be compelling.

Appendix A

Attempted Methods of Calibration

A number of experiments were made prior to settling on the final methods outlined in Sections A and B. These experiments, all performed with sheathed TCs from the same wire lot, are summarized below and in Figure A1.

STEP 1 TCs were wired together in a bundle with 6 TCs in a rectangular arrangement. The temperatures recorded among these TCs exhibited a range of 4.8 °C.

STEP 2 TCs were wired together in a bundle with 6 TCs surrounding the standard ("NIST") TC. The range of data was improved to ~3.5 °C, but a new problem arose, namely that the center TC was shielded by and thus was cooler than the 6 outside TCs.

STEP 3 TCs were wired together in a bundle with 6 TCs surrounding the standard TC. They were inserted into a 0.84 in. diameter by 2 in. deep hole bored into the center a 2 in. diameter by 4 in. long block of Haynes 188. In this setup, the data range was reduced to ~2.4 °C, but the outer TCs were again shielding the center TC. This problem vanished when the center TC was extended ¼ in. out of the bundle.

STEP 4 A new Haynes block with individual holes was then constructed. The first runs were made with the Haynes block resting on the bottom of the furnace tube. The reproducibility of this setup was excellent, and multiple runs were made and standard deviations of less than a degree were measured. However, the bottom positions were as much as 2 °C hotter than the top positions.

STEP 5 Another experiment was run in which two TCs from the same batch were placed in the center position but from opposite ends of the furnace, until the two TCs made contact. Despite the intimate contact, the two TCs showed a 6 °C difference. It is speculated that this difference is an immersion error [1] in which the heat is transferred differently down the length of the TCs due to temperature asymmetries along the length of the furnace. This method for TC arrangement should be avoided until it can be proven that both TCs read identically.

STEP 6 The setup in Step 4 was modified by centering the Haynes block in the tube furnace using ceramic boats (see also Figure 2). This setup produced slightly better standard deviations plus reduced the position bias values to ~1 °C. This setup was adopted as the final method.

Appendix B

Statistical Analyses of Data from the Metal Block Method of Calibrating Sheathed Thermocouples

Objective: Determine if positional biases relative to the center position exist at any of the six experimental positions in the metal block. If so, quantify them. Then quantify the 95% confidence intervals on the estimated bias in calibrating new thermocouples (TCs) against a standard TC, taking into account any positional biases that were determined.

Procedure: Select at random seven "identical" standard TCs and place them randomly into the seven positions in the metal block. Since the seven are assumed to be identical (and without bias as they are standard TCs), positional biases are simply estimated as $\Delta T = T_{TCi} - T_{TC0}$, where the subscripts indicate position in the metal block (Figure 2). Each experimental run generated a single estimate of the positional bias at each of the six experimental radial positions.

Data: Six experimental runs at each of three temperatures, 700 °C, 900 °C and 1100 °C, were conducted. Table BI below contains the raw experimental data.

Table BI
Table Entries are ΔT's by Position in the Metal Block and by Set Temperature °C

Temperature °C	Position 2	Position 3	Position 4	Position 5	Position 6	Position 7
700	1.83	2.03	1.69	1.62	1.64	2.13
700	1.08	1.77	0.95	0.69	0.94	1.43
700	0.80	1.11	0.81	0.32	0.29	1.03
700	0.73	0.97	0.12	0.58	0.82	0.63
700	1.20	1.45	0.91	0.43	0.84	1.00
700	1.46	1.40	1.05	0.87	1.11	1.33
900	2.01	1.95	1.37	1.29	1.37	2.11
900	1.11	1.69	0.63	0.44	0.57	1.32
900	1.07	1.23	0.67	0.19	0.05	0.98
900	1.10	1.12	0.10	0.48	0.85	0.95
900	1.37	1.47	0.70	0.27	0.65	1.20
900	1.49	1.45	0.85	0.73	0.91	1.41
1100	2.12	2.05	1.20	1.24	1.44	2.17
1100	0.99	1.74	0.45	0.61	0.43	1.35
1100	1.54	1.63	0.97	0.94	0.58	1.34
1100	1.27	1.27	0.33	0.76	1.09	1.19
1100	1.46	1.52	0.64	0.52	0.58	1.33
1100	1.50	1.52	0.79	0.77	0.72	1.32

Estimation of Mean Position i Bias with 95% Confidence Intervals:

Estimates of the position biases were calculated by taking the mean of the six experimental estimates of the positional bias as shown in equation (1) below. The estimation of the 95% confidence intervals on these estimated position biases is shown in equation (2) below. At the heart of equation (2) is a statistic, $S_{\Delta T}$, which is simply the standard deviation of the six trials pooled across positions. Prior to pooling, the standard deviations at the six experimental positions were found to be statistically insignificant and, thus, poolable. Notice in equation (2), the degrees of freedom associated with this pooled standard deviation was conservatively set at 6-1 = 5 to reflect the fact that there were only six experimental runs.

Eq. (1) $$\text{Estimated Position Bias at Position i} = \overline{\Delta T_i} = \sum_{j}^{6} \Delta T_{TCi} / 6$$

Eq. (2) $$\text{95\% Conf. Int. on Estimated Position Bias} = \overline{\Delta T_i} \pm t(0.975,5) S_{\Delta T}$$

Where $$S_{\Delta T} = \{\sum_{i}^{6} [\sum_{j}^{6} (\Delta T_{TCi} - \overline{\Delta T_i})^2 / 5] / 6\}^{1/2}$$

and the subscript j indicates the 6 experimental runs

Separate analyses were conducted for each of the three temperatures. The results of estimating position bias are given in Table BII below.

Table BII
Position Bias Results (in °C)

Position	700 °C ($S_{\Delta T}$ = 0.457)	900 °C ($S_{\Delta T}$ = 0.392)	1100 °C ($S_{\Delta T}$ = 0.330)
P2	1.18 ± 0.48	1.36 ± 0.41	1.48 ± 0.35
P3	1.46 ± 0.48	1.49 ± 0.41	1.62 ± 0.35
P4	0.92 ± 0.48	0.72 ± 0.41	0.73 ± 0.35
P5	0.76 ± 0.48	0.57 ± 0.41	0.81 ± 0.35
P6	0.94 ± 0.48	0.73 ± 0.41	0.81 ± 0.35
P7	1.26 ± 0.48	1.33 ± 0.41	1.45 ± 0.35

Procedure for Estimating Bias of New Experimental Thermocouple, TC:

Place a new experimental thermocouple, TC in position i (designation, TCi). Randomly select a standard thermocouple and place it in the center position (designation, STC0). Measure the temperature difference, $T_{TCi} - T_{STC0}$. Repeat N times. Calculate the Mean (TCi + Position i) Bias = $\Sigma (T_{TCi} - T_{STC0}) / N$. Lastly, subtract off the estimated mean position i bias from Table B above. These steps are summarized below.

Estimated Mean Bias for TCi = Mean(TCi + Position i) Bias - Mean Position i Bias

$$= \sum_{j=1}^{N} (T_{TCi} - T_{STC0}) / N - \overline{\Delta T_i}$$

Approximate 95% Confidence Interval on the Estimated Mean Bias for TCi

$$= \pm t(0.975,5) S_{Total}$$
$$= \pm t(0.975,5)[S^2_{TCi\ Bias} + S^2_{Position\ i\ Bias}]^{1/2}$$
$$\cong \pm t(0.975,5)[(S^2_{\Delta T} / N) + (\dot{S}^2_{\Delta T} / 6)]^{1/2}$$
$$= \pm 2.571[(1/N) + (1/6)]^{1/2} S_{\Delta T}$$

Table BIII below provides a summary of the approximate 95% confidence intervals on the estimated mean bias for calibrating a new TC in position i based on N trials.

Table BIII
Approximate 95% Confidence Intervals on the Estimated Mean Bias for TCi Based on N Trials (in °C)

Number of Trials	700 °C	900 °C	1100 °C
N = 1	±1.27	±1.09	±0.91
N = 2	±0.96	±0.82	±0.69
N = 3	±0.83	±0.71	±0.60
N = 5	±0.71	±0.61	±0.51

Additional Analyses:

An attempt was made to partition the positional biases into two groups, those holes above the horizontal axis of the metal block (P4, P5 and P6) and those below the horizontal axis of the metal block (P2, P3 and P7) (Figure 2). This grouping would greatly simplify the use of positional biases in future TC calibrations. At each of the three temperatures a statistical procedure known as a 1-way analysis of variance (ANOVA) was conducted for comparing the top three positional biases and again for comparing the bottom three positional biases. The results from these 1-way ANOVAs were that, in general:

a) the 3 top positions (P4, P5, P6) were statistically insignificant from one another.
b) the 3 bottom positions (P2, P3, P7) were statistically insignificant from one another.
c) the 3 top positions were significantly different, statistically from the 3 bottom positions.

It was assumed that there were no statistical differences across the three temperatures. Hence, the data were pooled across the three temperatures and then broken into two parts, the top positions and the bottom positions. Then, a 2-way ANOVA (with factors Temperature and Position) was conducted for each set of positions, top and bottom, separately. The results of each of these analyses found that the Temperature effect and the Position effect and the Temperature-x-Position interaction were not statistically significant. These results supported the conjecture that there were no differences across the three temperatures. The resultant estimated mean bias for the top positions was 0.78 and the estimated mean bias for the bottom positions was 1.40.

A components-of-variation analysis was conducted for each set of positions separately to quantify the variability in the response, ΔT, pooled over all three temperatures and all three positions within a group. The resultant $S_{\Delta T}$ statistic was statistically insignificant between the top and bottom positions and could themselves be pooled. The resultant grand pooled standard deviation of the response, ΔT, was $S_{\Delta T} = 0.416$ with 15 degrees of freedom. (Note: Even though there were 54 data points in each of the top and bottom pooled data sets, there were only six true repeats at each temperature resulting in five degrees of freedom for each of three temperatures. When pooled over the three temperatures, the total becomes 15 degrees of freedom. The results are summarized in Table BIV below.

Table BIV
Estimation of Mean Position Bias with 95% Confidence Intervals = $\Delta T_i \pm t(0.975,15)S_{\Delta T}/(18^{1/2})$

Position	Mean Bias with 95% Confidence Interval
Top (P4, P5, P6)	0.78 ± 0.21
Bottom (P2, P3, P7)	1.40 ± 0.21

Estimating Bias of New Experimental Thermocouple, TC:

The logic described above also applies here except that the formulas change slightly.

Estimated Mean Bias for TCi = Mean(TCi + Position i) Bias - Mean Position i Bias

$$= \sum_{j=1}^{N} (T_{TCi} - T_{STC0}) / N - \overline{\Delta T_i},$$

where, $\overline{\Delta T_i} = 0.775$ (Top) or 1.402 (Bottom)

Approximate 95% Confidence Intervals on the Estimated Mean Bias for TCi

$$
\begin{aligned}
&= \pm t(0.975,15)S_{Total} \\
&= \pm t(0.975,15)[S^2_{TCi\ Bias} + S^2_{Position\ i\ Bias}]^{1/2} \\
&\cong \pm t(0.975,15)[(S^2_{\Delta T}/N) + (S^2_{\Delta T}/18)]^{1/2} \\
&= \pm t(0.975,15)[(1/N) + (1/18)]^{1/2} S_{\Delta T} \\
&= \pm 2.131[(1/N) + (1/18)]^{1/2}(0.416)
\end{aligned}
$$

Table BV below provides a summary of the approximate 95% confidence intervals on the estimated mean bias for calibrating a new TC based on N trials.

Table BV
Approximate 95% Confidence Intervals on the Estimated Mean Bias for TCi Based on N Trials

Number of TCi Trials	N = 1	N = 2	N = 3	N = 5
95% Conf. Int.	±0.91	±0.66	±0.55	±0.45

Appendix C

Statistical Analyses of Data from the Metal Block Method of Calibrating Unsheathed Thermocouples

Objective: Determine if positional biases relative to the center position exist at any of the six experimental positions in the metal block. If so, quantify them. Then, quantify the 95% confidence intervals on the estimated bias in calibrating new thermocouples (TCs) against a standard TC, taking into account any positional biases that were discovered.

Procedure: Select at random seven "identical" standard TCs and place them randomly into the seven positions in the metal block. Since the seven are assumed to be identical (and without bias as they are standard TCs), positional biases are simply estimated as $\Delta T = T_{TCi} - T_{TC0}$, where the subscripts indicate position in the metal block. Each experimental run generated a single estimate of the positional bias at each of the six experimental positions around the outside of the metal block. On each run day, a single experimental run was conducted at each of five temperatures, 600 °C, 700 °C, 800 °C, 900 °C and 1000 °C.

Data: Seven experimental runs at each of five temperatures, 600 °C, 700 °C, 800 °C, 900 °C and 1000 °F were conducted. The response was $\Delta T = T_{TCi} - T_{TC0}$, i = 2, 3, 4, 5, 6, 7, where the i subscript indicates a given position in the metal block. The subscript i = 0 indicates the center or reference position. Table CI contains the raw experimental data.

Data Analysis:

At each of the five temperatures and for each of the six radial positions, both an estimate of the mean position bias and the run-to-run variation, $S_{\Delta T}$, were calculated as follows:

$$\overline{\Delta T} = \sum_{j=1}^{7} \Delta T_j / 7 = \text{Estimated mean position bias}$$

$$S_{\Delta T} = [\sum_{j=1}^{7} (\Delta T_j - \overline{\Delta T})^2 / (7-1)]^{1/2} = \text{Estimated run - to - run variation}$$

Where the j subscript represents the replicate experimental runs

The results of this initial effort at data reduction are given in Table CII.

Table CI

Table Entries are ΔT's by Position in the Metal Block and by Set Temperature °C

Temperature °C	**Position** P2	**Position** P3	**Position** P4	**Position** P5	**Position** P6	**Position** P7
600	0.20	0.03	-0.03	-0.11	-0.20	-0.14
600	0.03	0.25	0.73	-0.35	-0.35	-0.45
600	-0.20	0.07	0.28	-0.23	-0.50	-0.27
600	-0.12	0.50	-0.16	-0.34	0.04	-0.08
600	0.10	0.20	0.26	0.00	0.28	0.32
600	0.16	0.32	0.64	0.06	0.04	0.16
600	-0.20	0.26	0.10	-0.36	-0.20	-0.10
700	0.03	-0.08	-0.30	-0.47	-0.28	-0.18
700	0.28	0.12	0.43	-0.73	-0.47	-0.28
700	0.32	0.30	0.22	-0.33	-0.18	0.32
700	-0.17	0.58	-0.20	-0.37	-0.12	-0.15
700	0.00	0.27	0.27	-0.03	0.20	0.28
700	0.30	0.20	0.60	-0.30	-0.20	0.10
700	-0.34	0.68	-0.04	-0.66	0.16	-0.44
800	0.57	0.07	-0.18	-0.52	0.02	0.12
800	0.28	0.02	0.38	-0.67	-0.32	-0.05
800	0.54	0.20	0.29	-0.10	-0.09	0.57
800	0.04	0.39	-0.31	-0.50	0.00	-0.03
800	0.23	0.42	0.08	-0.15	0.32	0.32
800	1.10	1.10	0.80	-0.20	-0.10	-0.10
800	0.06	0.08	-0.12	-0.50	-0.38	-0.16
900	0.48	-0.12	-0.57	-0.50	-0.38	-0.28
900	0.18	0.07	0.39	-0.43	-0.31	0.28
900	0.91	0.83	0.64	-0.11	0.47	1.19
900	-0.04	0.66	-0.24	-0.58	-0.04	0.04
900	0.20	0.11	-0.40	-0.63	-0.05	0.24
900	0.30	0.30	1.20	-0.80	-0.10	0.20
900	0.56	0.44	-0.18	-0.32	-0.08	0.52
1000	0.20	0.31	-1.57	-1.34	-0.63	-0.60
1000	0.85	0.59	0.86	0.16	0.06	0.61
1000	1.43	1.09	0.71	0.47	0.36	0.86
1000	0.05	1.22	-0.97	-0.47	0.23	-0.55
1000	0.60	0.66	0.06	-0.46	0.73	0.20
1000	0.22	0.66	0.12	-0.42	-0.32	0.66
1000	0.56	0.39	0.17	-0.68	0.27	0.52

Table CII

Estimates of Mean Position Bias, $\overline{\Delta T}$, (Top Entry) and Run - to - Run Variation, $S_{\Delta T}$ (Bottom Entry)

Temperature °C	**Position** P2	**Position** P3	**Position** P4	**Position** P5	**Position** P6	**Position** P7
600	-0.00 0.17	0.23 0.16	0.26 0.33	-0.19 0.18	-0.13 0.27	-0.08 0.26
700	0.06 0.26	0.30 0.26	0.14 0.33	-0.41 0.24	-0.13 0.24	-0.05 0.29
800	0.40 0.37	0.32 0.38	0.13 0.39	-0.38 0.22	-0.08 0.23	0.10 0.26
900	0.37 0.31	0.33 0.34	0.12 0.64	-0.48 0.22	-0.07 0.27	0.31 0.46
1000	0.56 0.47	0.70 0.34	-0.09 0.88	-0.39 0.59	0.10 0.45	0.24 0.59

Error Analysis:

Next, an investigation into the error structure was conducted to determine if the errors were stable over all six positions and over all five temperatures. While the run-to-run variations did not change significantly from one position to the next, they did change significantly across the five temperatures. In order to quantify the run-to-run variation, an unreplicated, two-way ANOVA was conducted with factors Position and Run Day, for each of the five temperatures separately. The variances due to Run Day and the Run Day*Position interaction were estimated and combined to give an overall estimate of the run-to-run variation. A Satterthwaite formula was applied to estimate the approximate degrees of freedom that could be assigned to the combined error estimates. Table CIII below shows the estimated test errors with estimated degrees of freedom for each of the five temperatures.

Table CIII

Estimates of Test Errors for Run-to-Run Variation, $S_{\Delta T}$ (in °C)

Temperature °C	$S_{\Delta T}$ = Estimated Test Error (i.e., Run-to-Run Variation)	ν = Estimated Degrees of Freedom
600	0.23	34.9
700	0.27	35.8
800	0.32	29.3
900	0.40	25.5
1000	0.58	18.9

To complete the error analysis, a regression model was fit that quantified how the run-to-run variations changed with temperature. The resultant estimated regression fit was: $S_{\Delta T} = 0.2106 + 0.0006315e^{(T/157.1)}$ and is shown graphically in Figure C1. One can use this regression equation to predict the test error at a specified temperature with approximately 29 degrees of freedom, the average of the estimated degrees of freedom in Table CIII.

Position Bias Analysis:

Next, a two-way ANOVA with factors Position and Temperature was conducted to determine if there were significant differences in ΔT among the six positions or among the five temperatures. While there were no significant Temperature effects nor any significant Position*Temperature interactions, there were highly significant differences among the Positions. Hence, a follow-up one-way ANOVA and Bonferroni multiple comparison procedure (MCP) with factor = Position were run to quantify how the positions were different. The results are summarized in Figure C2, the Bonferroni MCP graph. The positions with MCP error bars that do not overlap are significantly different, statistically. Hence, the mean ΔT for position P5 is significantly lower than the other five positions. The mean ΔT for position P6 is significantly lower than both position P2 and position P3. Officially, there are no other statistical significances in mean ΔT among the six positions.

Table CIV summarizes the mean position bias and approximate 95% confidence intervals on these mean position biases. For each position, the estimated mean position biases were calculated by averaging the mean position biases over the five temperatures as it was determined that the difference was statistically insignificant over temperature. In other words, the estimated mean position bias does not change over temperature. The approximate 95% confidence intervals on these estimated mean position biases were calculated as follows:

$$\sim 95\%\ \text{Conf. Int.} = \overline{\overline{\Delta \mathrm{T}}} \pm t(0.975,29) S_{\Delta T} / (7^{1/2})$$

Where: $\overline{\overline{\Delta \mathrm{T}}}$ is the estimated mean position bias averaged over both replicate run days and temperature

$S_{\Delta T} = 0.2106 + 0.0006315 e^{(T/157.1)}$ is the estimated run-to-run variation

7 is the number of replicate run days used in the estimate of $S_{\Delta T}$

Notice that while the run-to-run variation, $S_{\Delta T}$, changed with temperature, it did not change from position to position.

Table CIV
Mean Position Bias with Approximate 95% Confidence Interval (in °C)

Position	600 °C	700 °C	800 °C	900 °C	1000 °C
P2	0.28 ± 0.19	0.28 ± 0.21	0.28 ± 0.24	0.28 ± 0.31	0.28 ± 0.45
P3	0.38 ± 0.19	0.38 ± 0.21	0.38 ± 0.24	0.38 ± 0.31	0.38 ± 0.45
P4	0.11 ± 0.19	0.11 ± 0.21	0.11 ± 0.24	0.11 ± 0.31	0.11 ± 0.45
P5	-0.37 ± 0.19	-0.37 ± 0.21	-0.37 ± 0.24	-0.37 ± 0.31	-0.37 ± 0.45
P6	-0.06 ± 0.19	-0.06 ± 0.21	-0.06 ± 0.24	-0.06 ± 0.31	-0.06 ± 0.45
P7	0.10 ± 0.19	0.10 ± 0.21	0.10 ± 0.24	0.10 ± 0.31	0.10 ± 0.45

Procedure for Estimating Bias of a New Experimental Thermocouple, TC:

Place a new experimental thermocouple, TC in position i (designation, TCi). Randomly select a standard TC and place it in the center position. Measure the temperature difference, ΔTCi. Repeat j = 1, …, N times. Calculate the Mean (TC + Position i) Bias = $\Sigma(\Delta TCi)_j$ / N, where the summing operation is over the repeat measurements (i.e. over the j subscript, not the i positional subscript). Lastly, subtract the estimated mean position i bias from Table CIV above. These steps are summarized below.

Estimated Mean Bias for TCi = Mean(TCi + Position i) Bias - Mean Position i Bias

$$= \sum_{j=1}^{N} (\Delta TCi)_j / N - \overline{\Delta T_i}$$

Approximate 95% Confidence Interval on the Estimated Mean Bias for TCi

$$\begin{aligned}
&= \pm t(0.975,29) S_{Total} \\
&= \pm t(0.975,29)[S^2_{TCi\ Bias} + S^2_{Position\ i\ Bias}]^{1/2} \\
&\cong \pm t(0.975,29)[(S^2_{\Delta T} / N) + (S^2_{\Delta T} / 7)]^{1/2} \\
&= \pm 2.045[(1/N) + (1/7)]^{1/2} S_{\Delta T} \\
&= \pm 2.045[(1/N) + (1/7)]^{1/2} [0.2106 + 0.0006315 e^{(T/157.1)}]
\end{aligned}$$

Table CV provides a summary of the approximate 95% confidence intervals in the estimated mean bias for calibrating a new TC based on N trials. Note that these 95% confidence intervals do not change as a function of position i around the metal block, but only as a function of temperature.

Table CV
Approximate 95% Confidence Intervals on the Estimated Mean Bias for a New TC Based on N Trials (in °C)

N = # of Trials	T = 600 °C	T = 700 °C	T = 800 °C	T = 900 °C	T = 1000 °C
1	± 0.56	± 0.62	± 0.73	± 0.95	± 1.35
2	± 0.32	± 0.35	± 0.41	± 0.53	± 0.76
3	± 0.23	± 0.26	± 0.31	± 0.39	± 0.56
5	± 0.17	± 0.19	± 0.22	± 0.28	± 0.41

Appendix D

Statistical Analyses of Data from the Tying Method of Calibrating Unsheathed Thermocouples

Objective: Quantify the 95% confidence intervals on the estimated bias in calibrating new unsheathed thermocouples (TCs) against a standard TC using the tying method of calibration.

Procedure: Four “identical” standard TCs (identified as B6, B7, B8 and B9) were selected for this study. TC B6 was designated as the reference standard to which each of the other three were compared (i.e., calibrated against). The four TCs were tied together according to the calibration procedure and subjected to five separate temperatures (600 °C, 700 °C, 800 °C, 900 °C and 1000 °C) each day of testing. There were five test days in all.

Data: At each of five temperatures, five replicate measurements, each taken on a different day, were made on each of the four TCs. The response calculated was $\Delta T = T_{Bi} - T_{B6}$, $i = 7, 8, 9$, where the Bi indicate the non-reference TCs. Table DI below contains the raw experimental data. Note that only four replicates were conducted at 600 °C.

Table DI
Table Entries are ΔT’s by TC (B7, B8, B9) and by Set Temperature °C

Temperature °C	B7	B8	B9
600	-0.08	-0.15	-0.08
600	0.53	0.00	0.35
600	-0.24	-0.08	-0.02
600	0.20	0.14	0.22
700	-0.33	-0.22	-0.18
700	-0.18	-0.13	-0.25
700	0.55	0.05	0.48
700	-0.06	0.00	0.08
700	-0.16	0.00	-0.02
800	-0.23	0.05	0.12
800	-0.43	-0.25	-0.45
800	0.55	0.18	0.43
800	0.08	0.00	0.26
800	-0.48	-0.22	-0.38
900	-0.25	-0.02	0.00
900	-0.45	-0.30	-0.45
900	0.65	0.28	0.38
900	0.08	0.02	0.32

Table DI (Concluded)

900	-0.46	-0.06	-0.34
1000	-0.08	0.18	0.13
1000	-0.53	-0.10	-0.50
1000	0.75	0.25	0.60
1000	0.34	0.12	0.48
1000	-0.66	-0.28	-0.42

Error Analysis:

At each of the five temperatures and for each of the three non-reference TCs that were tied together, an estimate of the run-to-run variation in the estimated bias, $S_{\Delta T}$, was calculated as follows:

$$S_{\Delta T} = [\sum_{j=1}^{5} (\Delta T_j - \overline{\Delta T})^2 /(5-1)]^{1/2} = \text{Estimated run - to - run variation}$$

Where $\overline{\Delta T} = \sum_{j=1}^{5} \Delta T_j / 5 = \text{Estimated mean bias in TC Bi}$

and the j subscript represents the replicate experimental runs

The results are given in Table DII below.

Table DII
Estimates of Mean TC Bi Bias, $\overline{\Delta T}$, (Top Entry) and Run - to - Run Variation, $S_{\Delta T}$ (Bottom Entry)

Temperature °C	B7	B8	B9
600	0.10 0.34	-0.02 0.12	0.12 0.20
700	-0.04 0.34	-0.06 0.11	0.02 0.29
800	-0.10 0.43	-0.05 0.18	-0.01 0.39
900	-0.09 0.47	-0.02 0.21	-0.02 0.37
1000	-0.04 0.59	0.04 0.22	0.06 0.50

While the run-to-run variations ($S_{\Delta T}$'s) did not change significantly from one TC to the next, they did change significantly across the five temperatures. Hence, at each of the five temperatures an unreplicated, two-way ANOVA was conducted with factors TC and Run Day. The variances due to Run Day and the Run Day∗TC interaction were estimated and combined to give an overall estimate of the run-to-run variation, $S_{\Delta T}$. A Satterthwaite formula was applied to estimate the approximate degrees of freedom that could be assigned to the combined error estimates. Table DIII below shows the estimated run-to-run variations, $S_{\Delta T}$, with their estimated degrees of freedom for each of the five temperatures.

Table DIII
Run-to-Run Variation Pooled Over TC as a Function of Temperature (in °C)

Temperature °C	$S_{\Delta T}$	ν = Estimated Degrees of Freedom
600	0.24	6.6
700	0.27	7.6
800	0.35	6.3
900	0.37	6.5
1000	0.47	6.5

To complete the error analysis, a regression model was fit that quantified how the run-to-run variations, $S_{\Delta T}$, changed with temperature. The resultant estimated regression fit was $S_{\Delta T} = -0.110 + 0.000577$(Set Temperature) and is shown graphically in Figure D1. One can use this regression equation to predict the run-to-run variation, $S_{\Delta T}$, at a specified temperature with approximately 6.7 degrees of freedom, the average of the estimated degrees of freedom in Table DIII above.

Estimating Bias of a New Experimental Thermocouple, TC:

1. Tie the new TC to be calibrated with a standard TC. Measure the temperature difference, ΔT_j. Repeat $j = 1, \ldots, N$ times.

2. Calculate the mean and standard deviation (Std. Dev.) of the TC bias measurements as shown below.

$$\overline{\Delta T} = \sum_{j=1}^{N} (\Delta T_j)/N = \text{Estimated Mean of TC Bias Measurements}$$

$$S_{\Delta T\_TC} = [\sum_{j=1}^{N} (\Delta T_j - \overline{\Delta T})^2/(N-1)]^{1/2} = \text{Estimated Std. Dev. of TC Bias Measurements}$$

3. Compare $S_{\Delta T\_TC}$ to the predicted value of $S_{\Delta T} = -0.110 + 0.000577$(Set Temperature) using the standard F-test for comparing two standard deviations: $F = (S_{Larger} / S_{Smaller})^2$.

4. If the F-test is not significant, pool $S_{\Delta T\_TC}$ and the predicted $S_{\Delta T}$ to achieve a pooled $S_{\Delta Tp}$ with N-1+6.7 degrees of freedom as follows:

$$S_{\Delta Tp} = \{[(N-1)(S_{\Delta T\_TC})^2 + (6.7)(\text{predicted } S_{\Delta T})^2] / (N-1+6.7)\}^{1/2}$$

5. Calculate the approximate 95% confidence interval on the estimated mean bias for TC as follows:

$$\sim 95\% \text{ Conf. Int. on Estimated Mean Bias for TC} = \overline{\Delta T} \pm t(0.975, N-1+6.7) S_{\Delta Tp} / N^{1/2}$$

Table DIV below shows the approximate 95% confidence intervals on the estimated mean bias for the five temperatures studied and for N = 1, 2, 3, 4 and 5 repeat measurements of the new TC bias. It also gives the appropriate values for the t-factor, t(0.975,N-1+6.7). Note the table entries assume that the pooled estimate of the error, $S_{\Delta Tp}$, is equal to the predicted $S_{\Delta T}$.

Table DIV
Approximate 95% Confidence Intervals on the Estimated Mean Bias Based on N Trials

	t(0.975,N-1+6.7)	T = 600 °C	T = 700 °C	T = 800 °C	T = 900 °C	T = 1000 °C
$S_{\Delta Tp}$		0.24	0.29	0.35	0.41	0.47
N = 1	2.39	±0.56	±0.70	±0.84	±0.98	±1.11
N = 2	2.32	±0.39	±0.48	±0.58	±0.67	±0.77
N = 3	2.27	±0.31	±0.39	±0.46	±0.54	±0.61
N = 4	2.24	±0.26	±0.33	±0.39	±0.46	±0.52
N = 5	2.21	±0.23	±0.29	±0.35	±0.40	±0.46

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6. M.P.E. Desvaux, in Measurement of High Temperature Mechanical Properties of Materials, eds. M.S. Loveday, M.F. Day, and B.F. Dyson, Her Majesty's Stationary Office, London, 1981, p. 96.
7. Private communication with R. Park, Marlin Manufacturing Corp, 2000.
8. A.I. Dahl, in Temperature, its Measurement and Control in Science and Industry, Vol. 1, Reinhold Publishing Corp., NY, 1941, p. 1238.

Figure 1. Test setup for calibrating thermocouples.

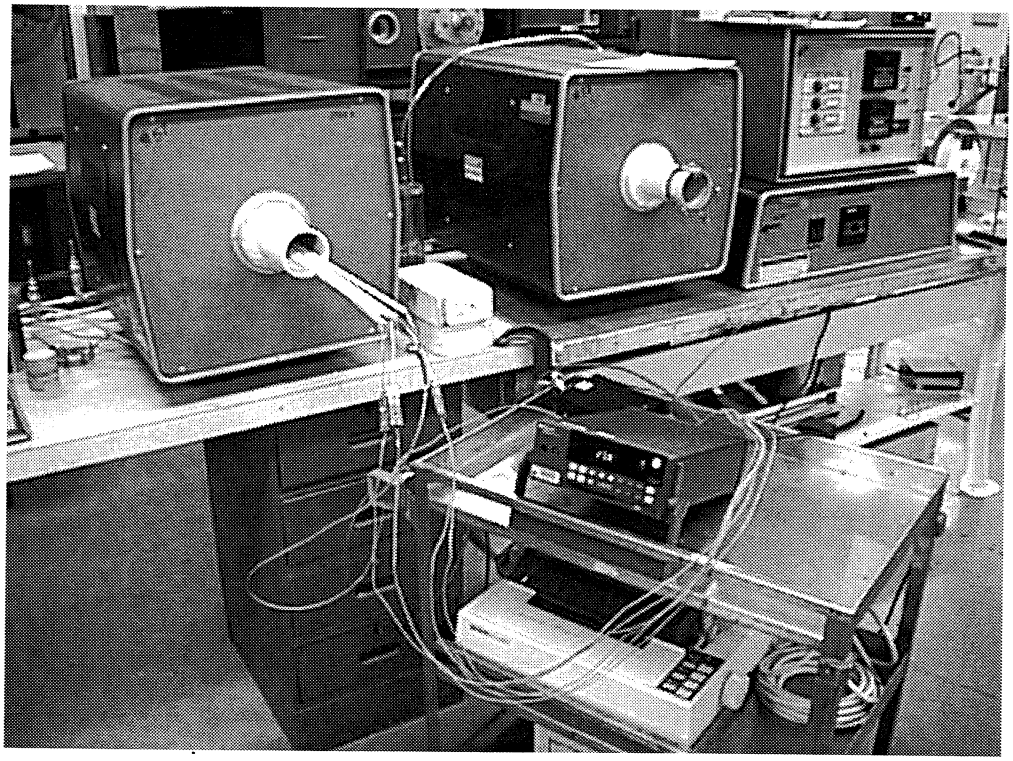
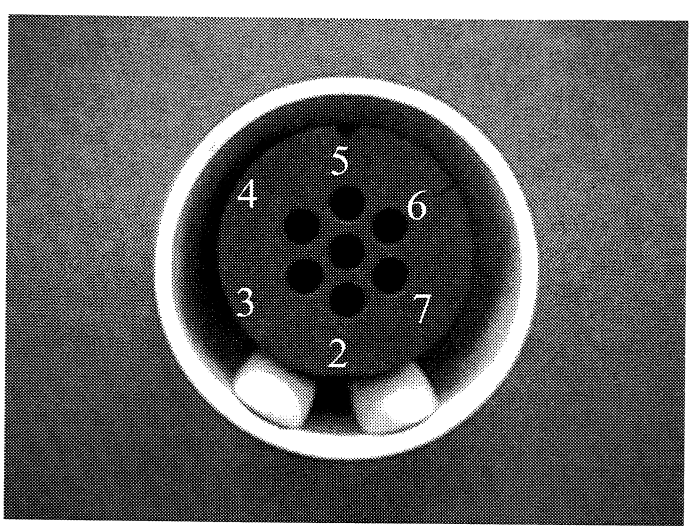


Figure 2. The 2 in. diameter Haynes 188 block (left) on ceramic boats inside of furnace tube for calibrating sheathed TC. The 1 in. diameter Haynes 188 block (right) on furnace tube for calibrating unsheathed TC. The TC positions are numbered.



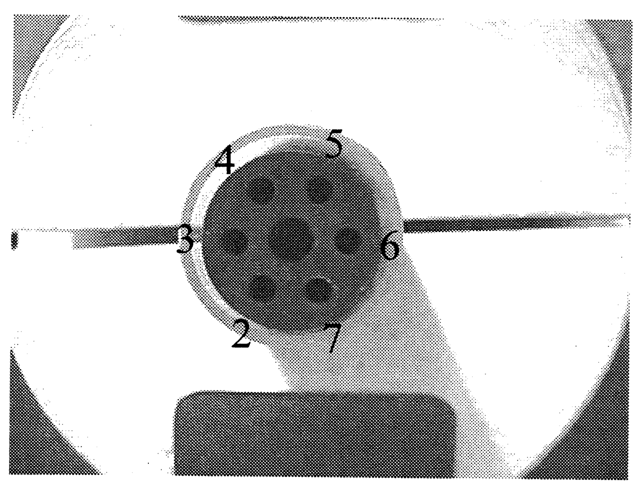


Figure 3. Tying method for unsheathed TC's.

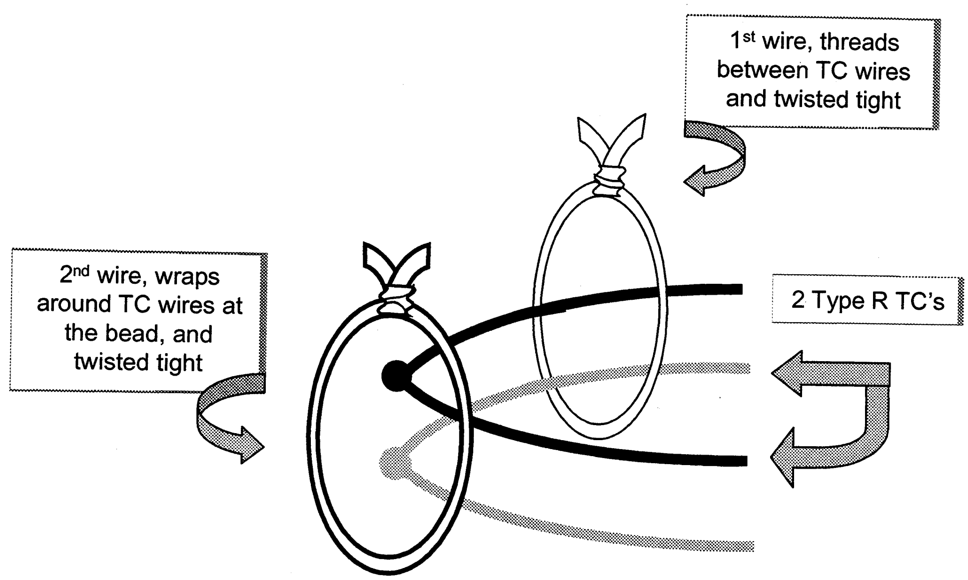


Figure 4a. 95% Confidence Intervals for Type R Thermocouple Calibrations at 600 °C.

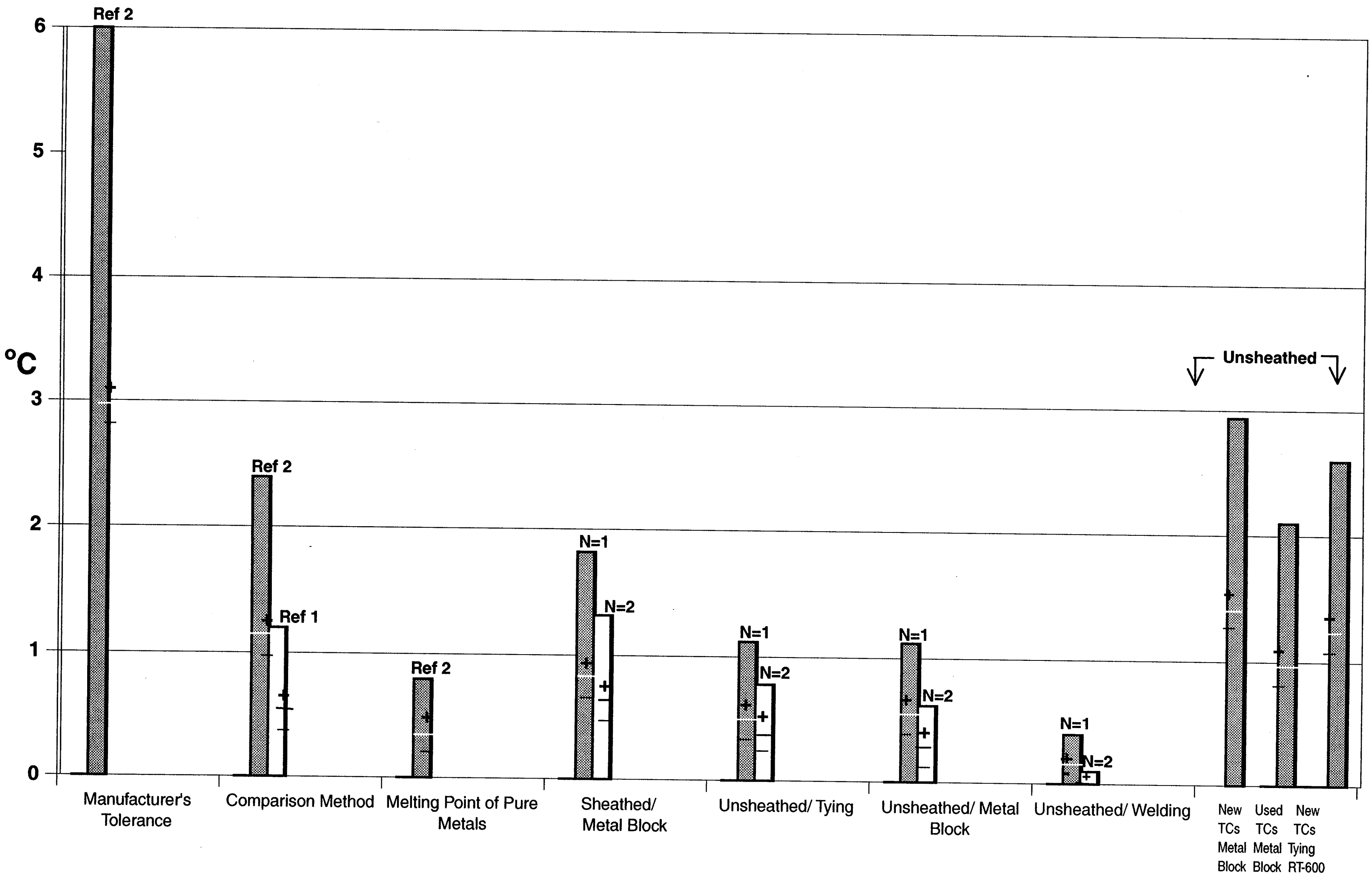


Figure 4b. 95% Confidence Intervals for Type R Thermocouple Calibrations at 1000 °C.

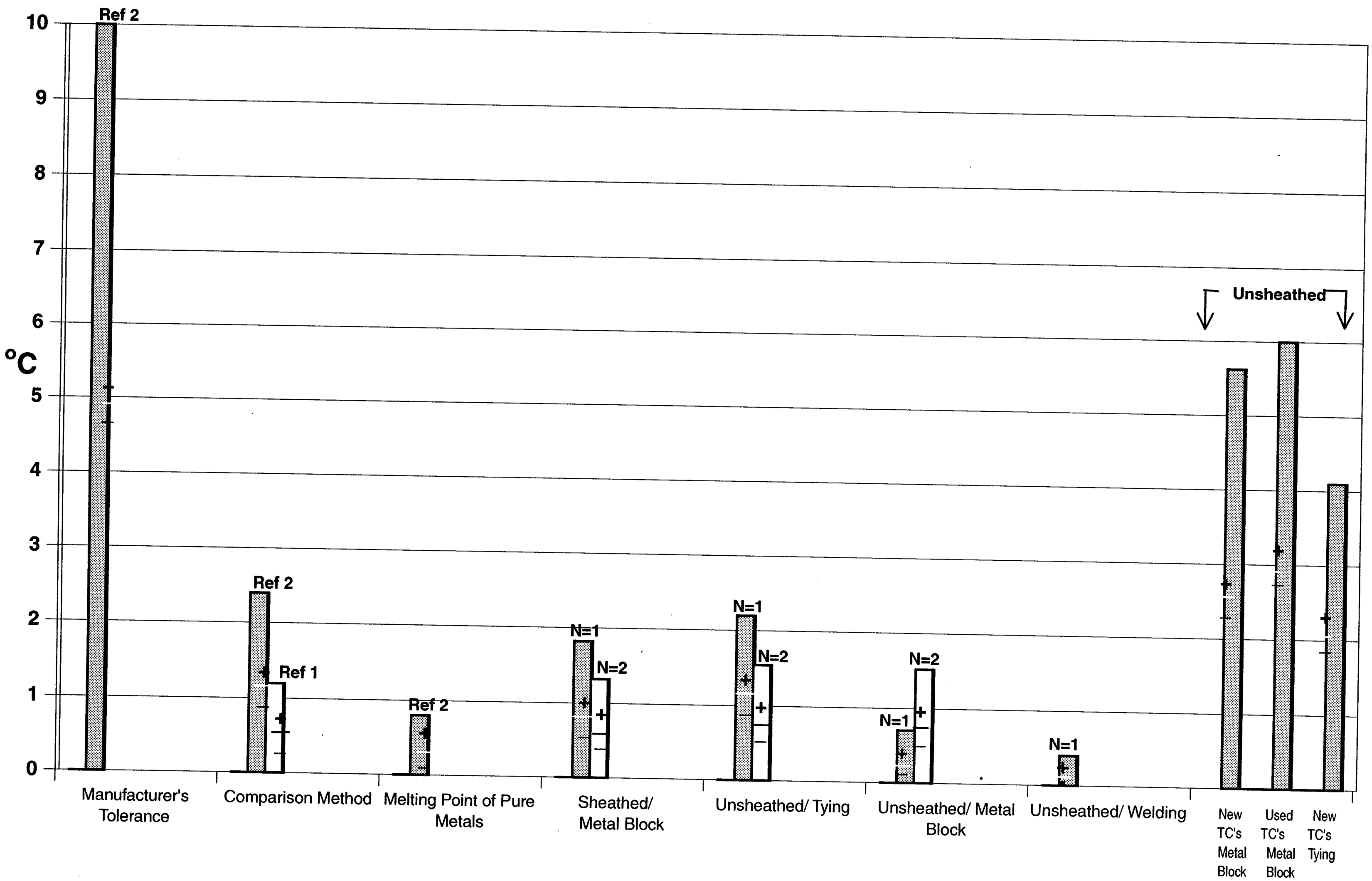


Figure 5. Calibration data on 7 batches (s/n in parenthesis) of Type R thermocouples from Marlin Manufacturing Co.

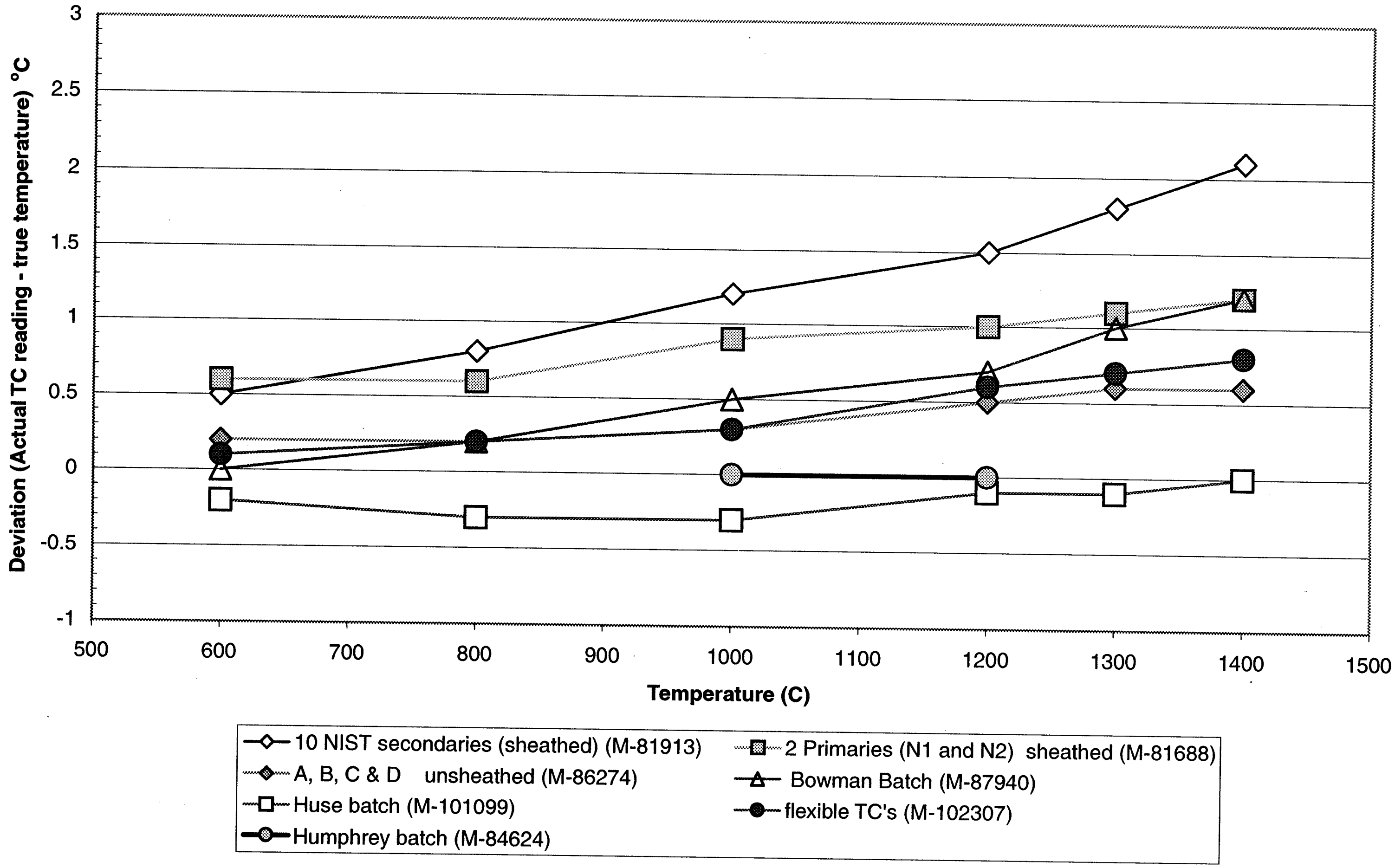


Figure 6. Calibration drift during isothermal exposure in air of Type K thermocouples. Data is from Dahl [8] for a wire diameter of 18 gage = 0.040 inches.

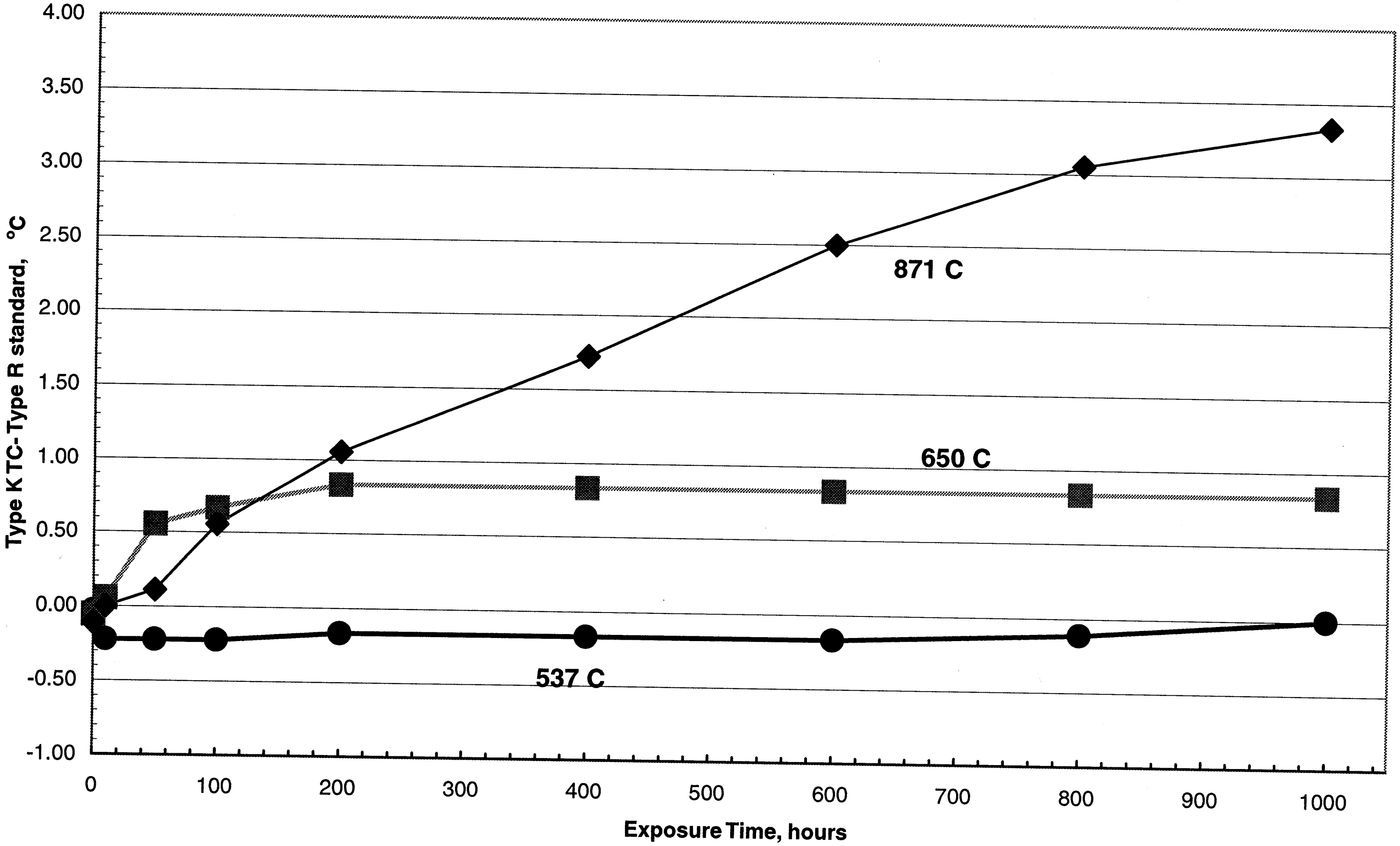


Figure 7. Stability of unsheathed Type K thermocouples (0.012 in. wire diameter) in air.

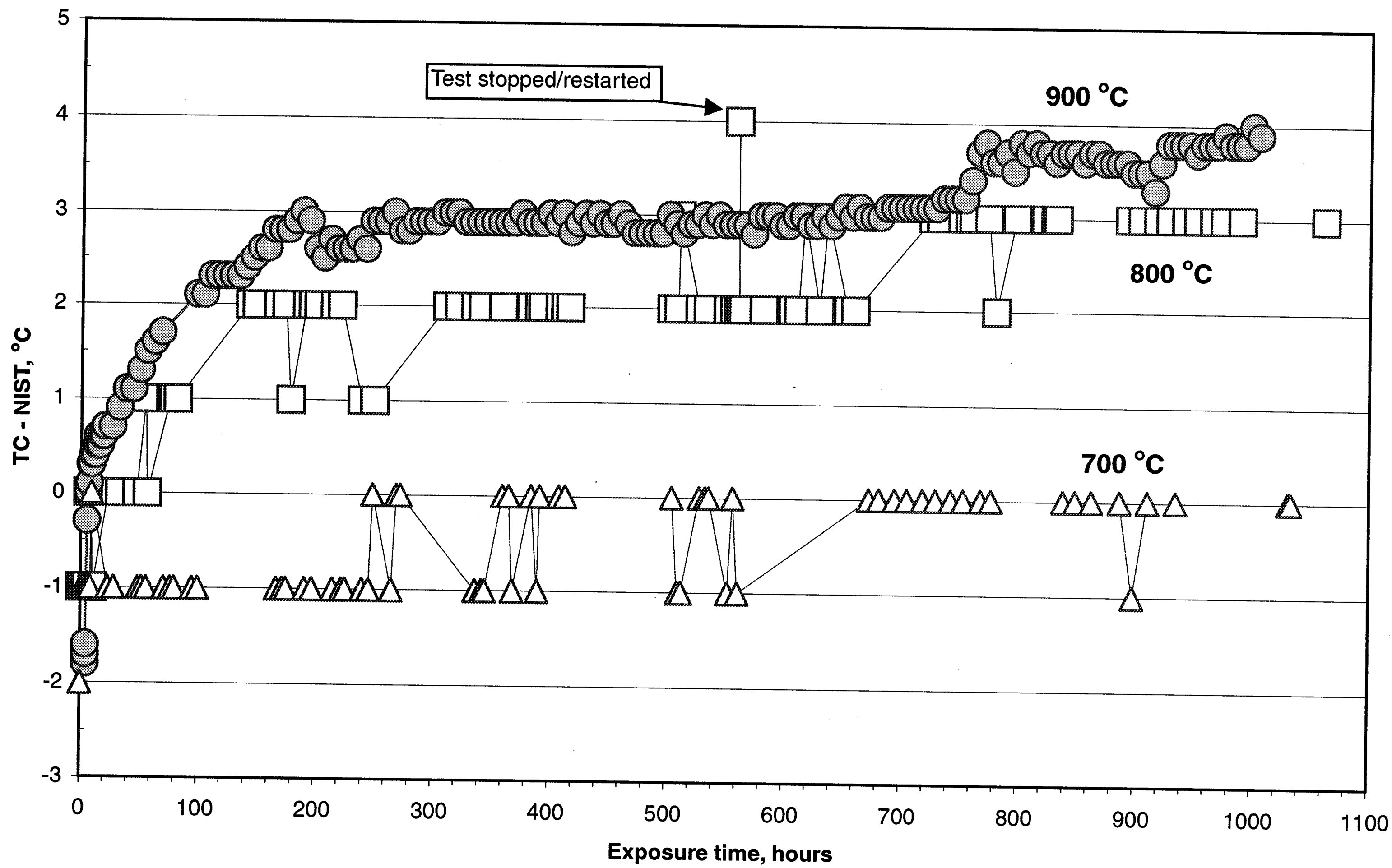


Figure 8. Comparison of Type K calibration drift from the current study at 800 and 900 °C and that of Dahl [8] at 871 °C. Note symbol size is proportional to wire diameter.

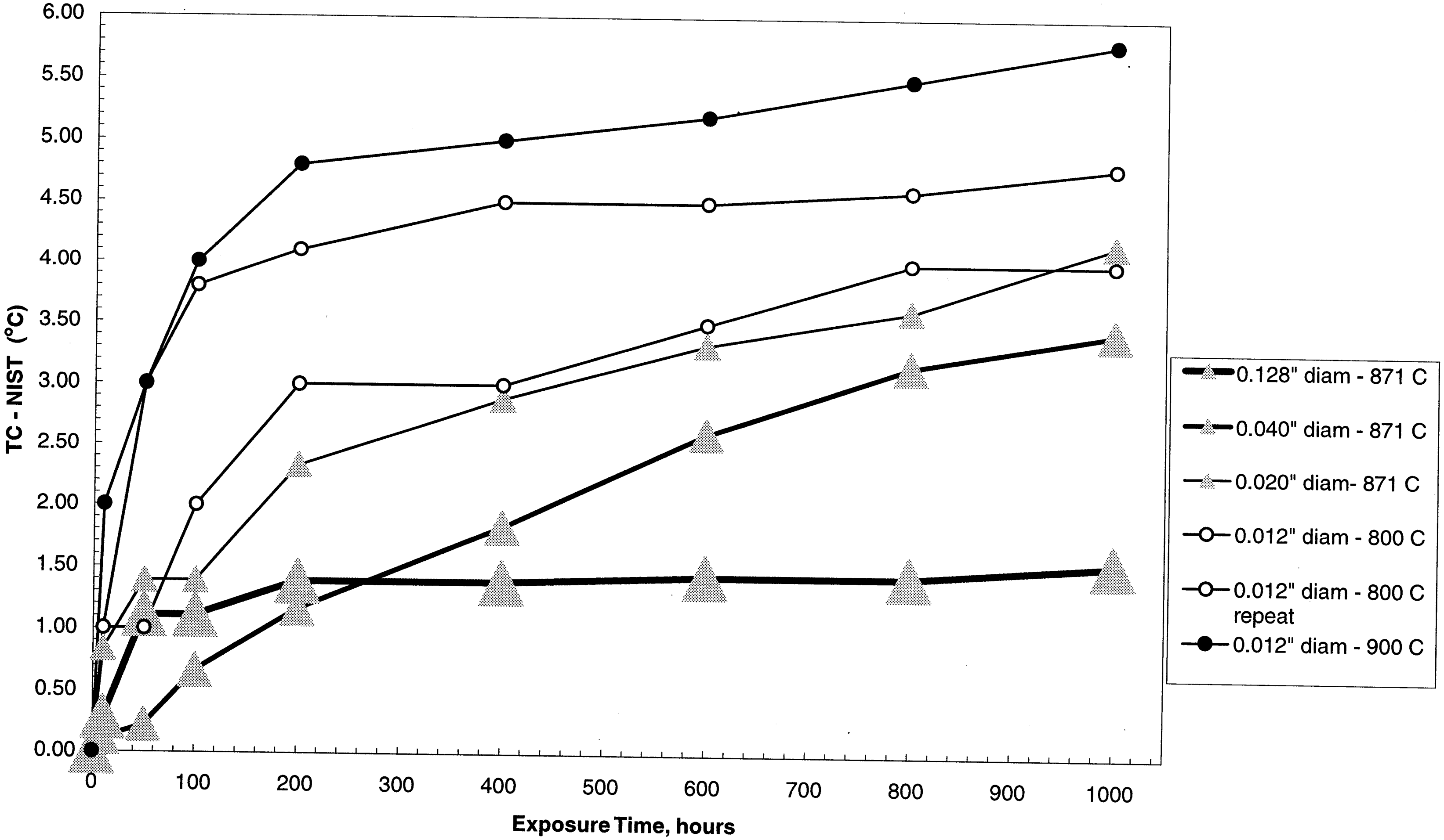


Figure 9a. Stability in air of Type K thermocouples at 800 °C.

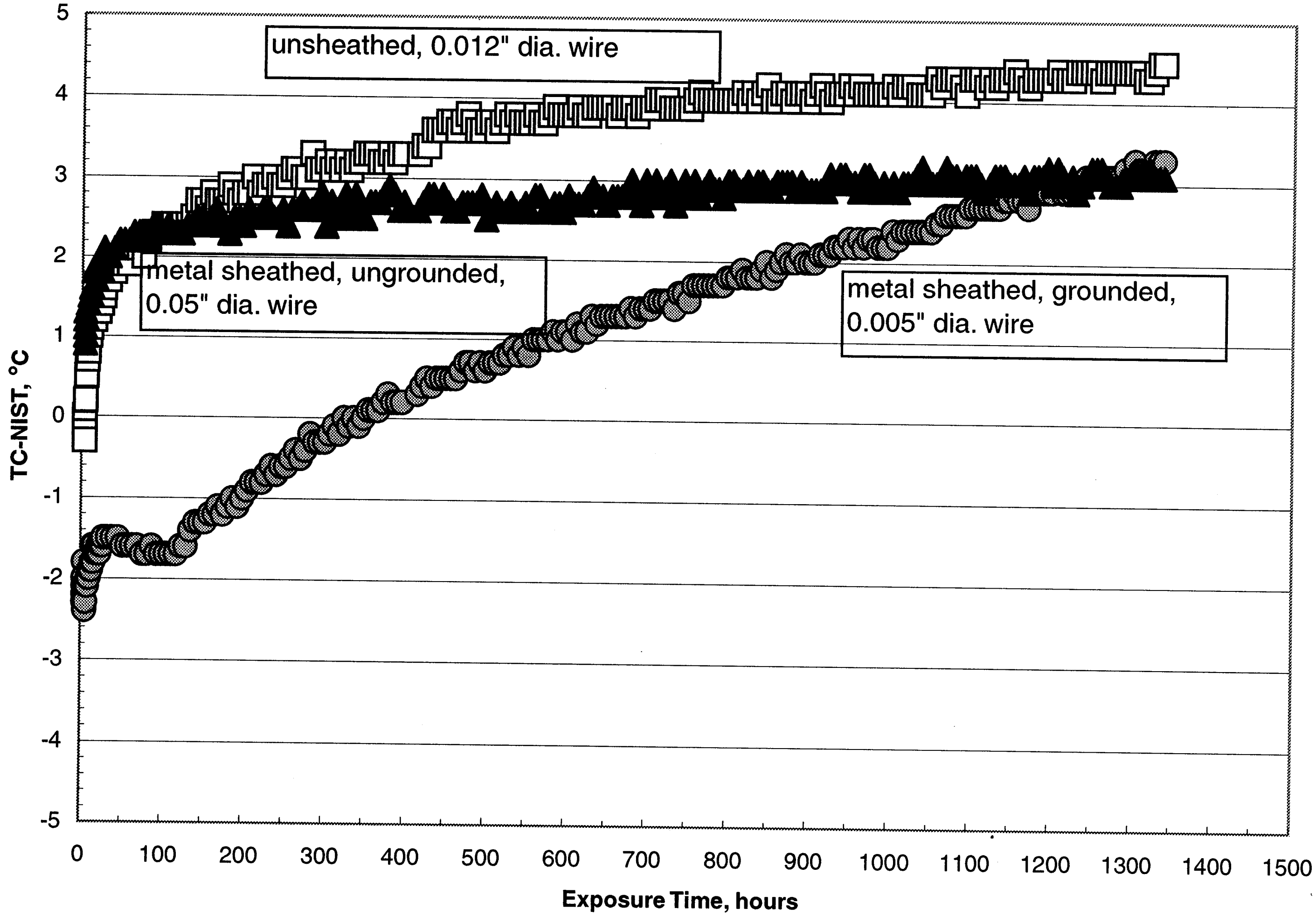


Figure 9b. Reproducibility of Type K thermocouple stability at 800 °C. Repeat of the experiment shown in Fig. 9a, with the exception that the first 12 hours of exposure data is missing.

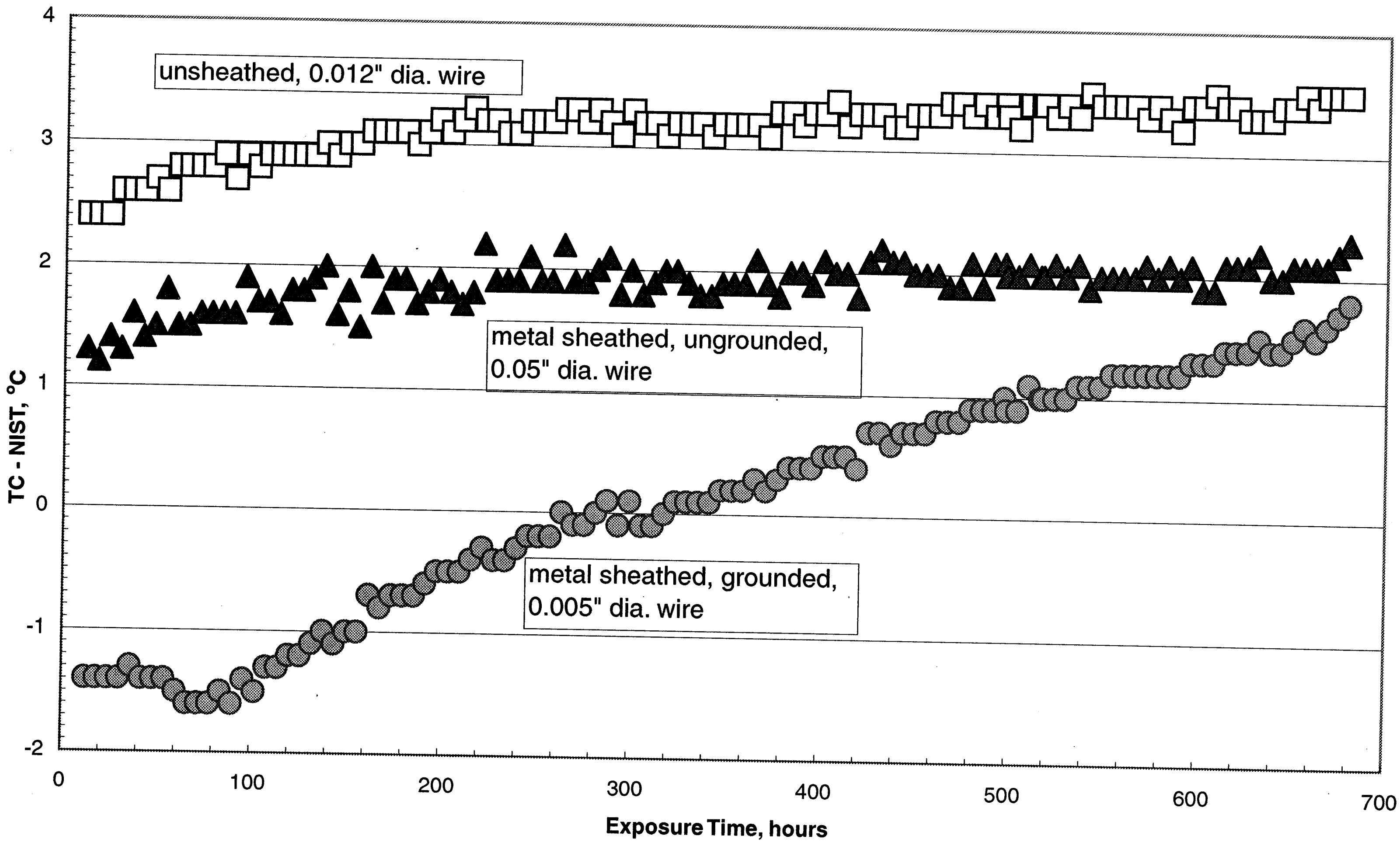


Figure 10. Stability in air of Type K thermocouples at 900 °C.

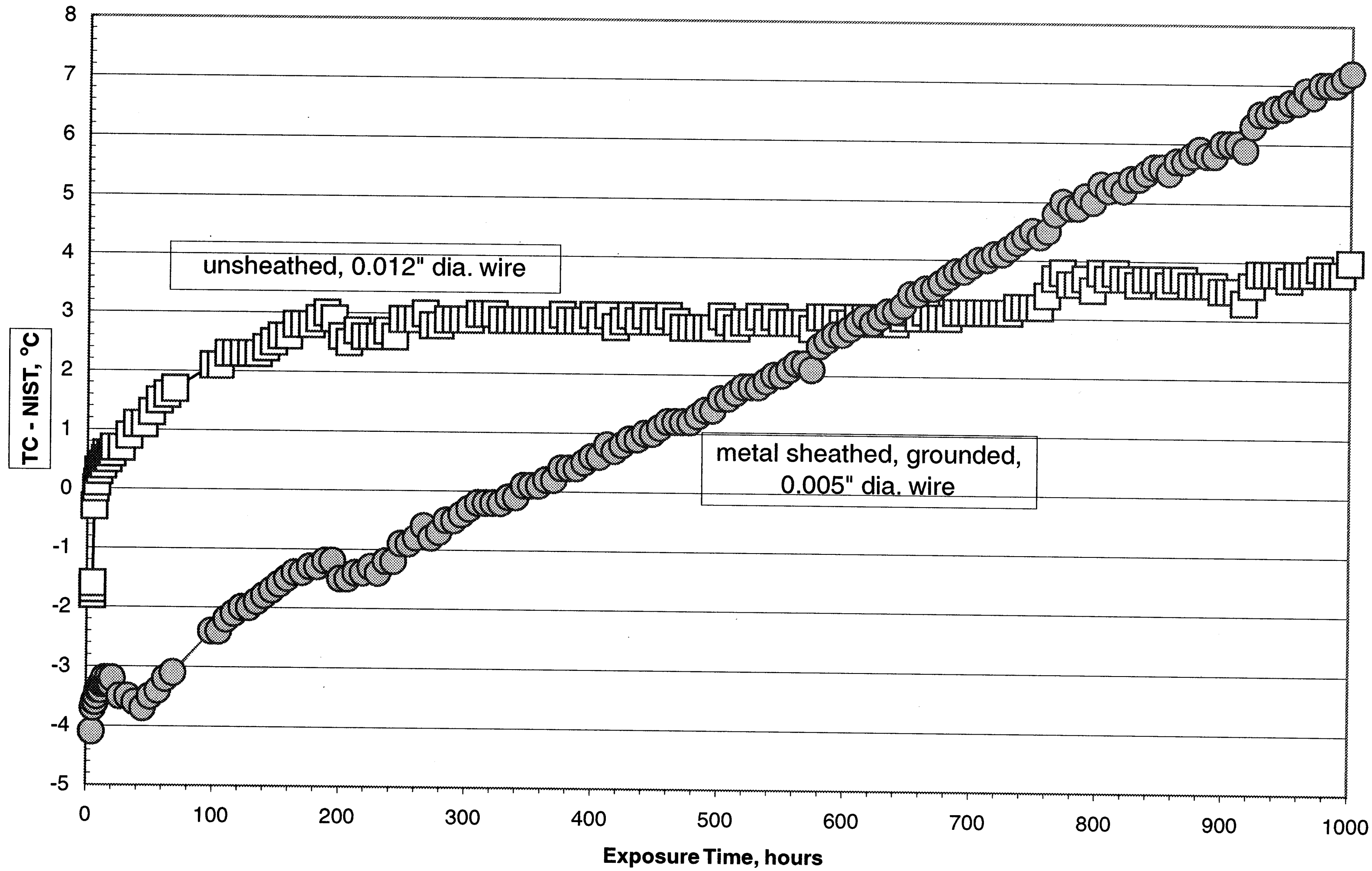


Figure 11a. Comparison of Accuracy and Stability for Type R and Type K TC's at 700 °C.

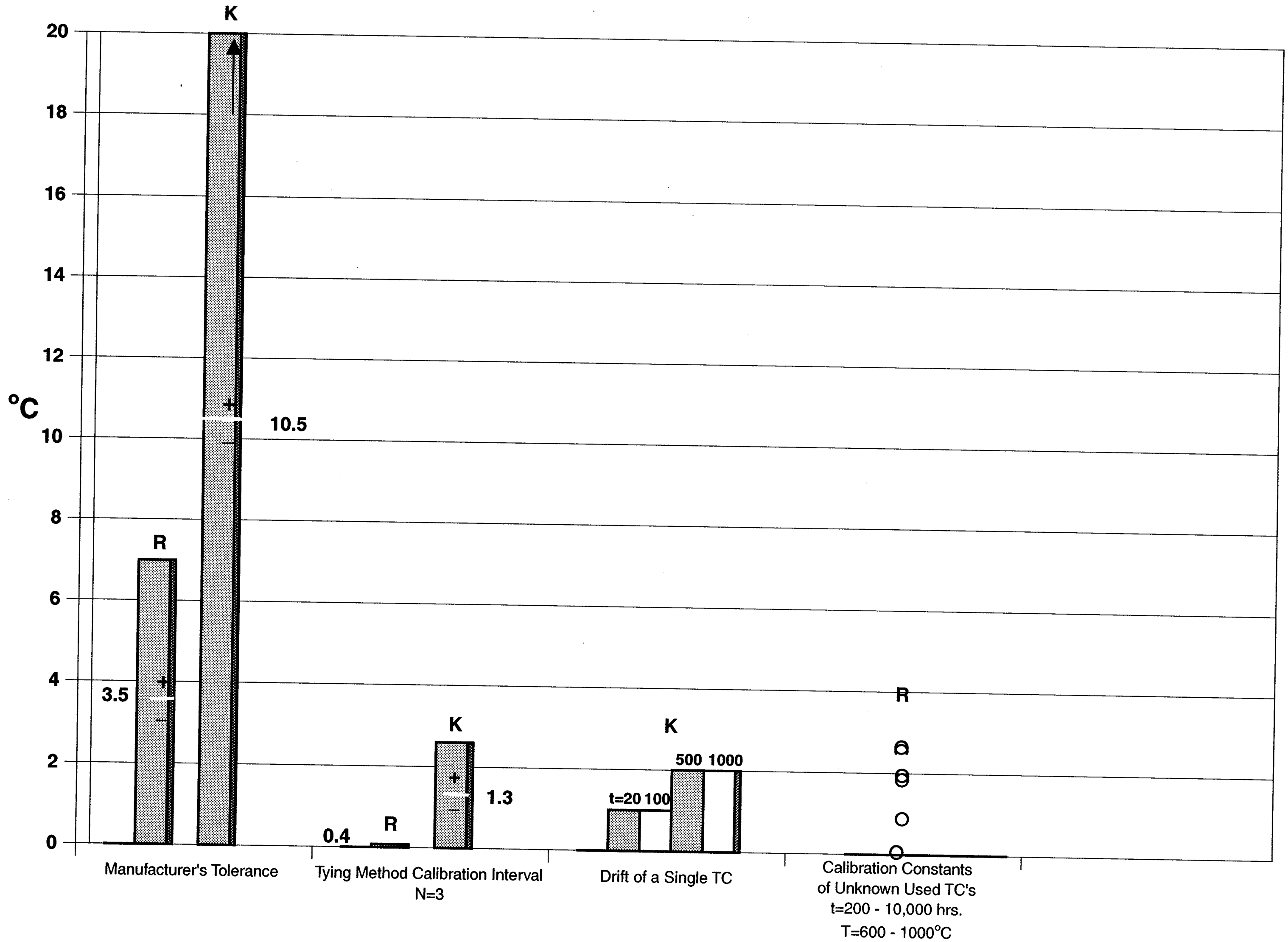


Figure 11b. Comparison of Accuracy and Stability for Type R and Type K TC's at 900 °C.

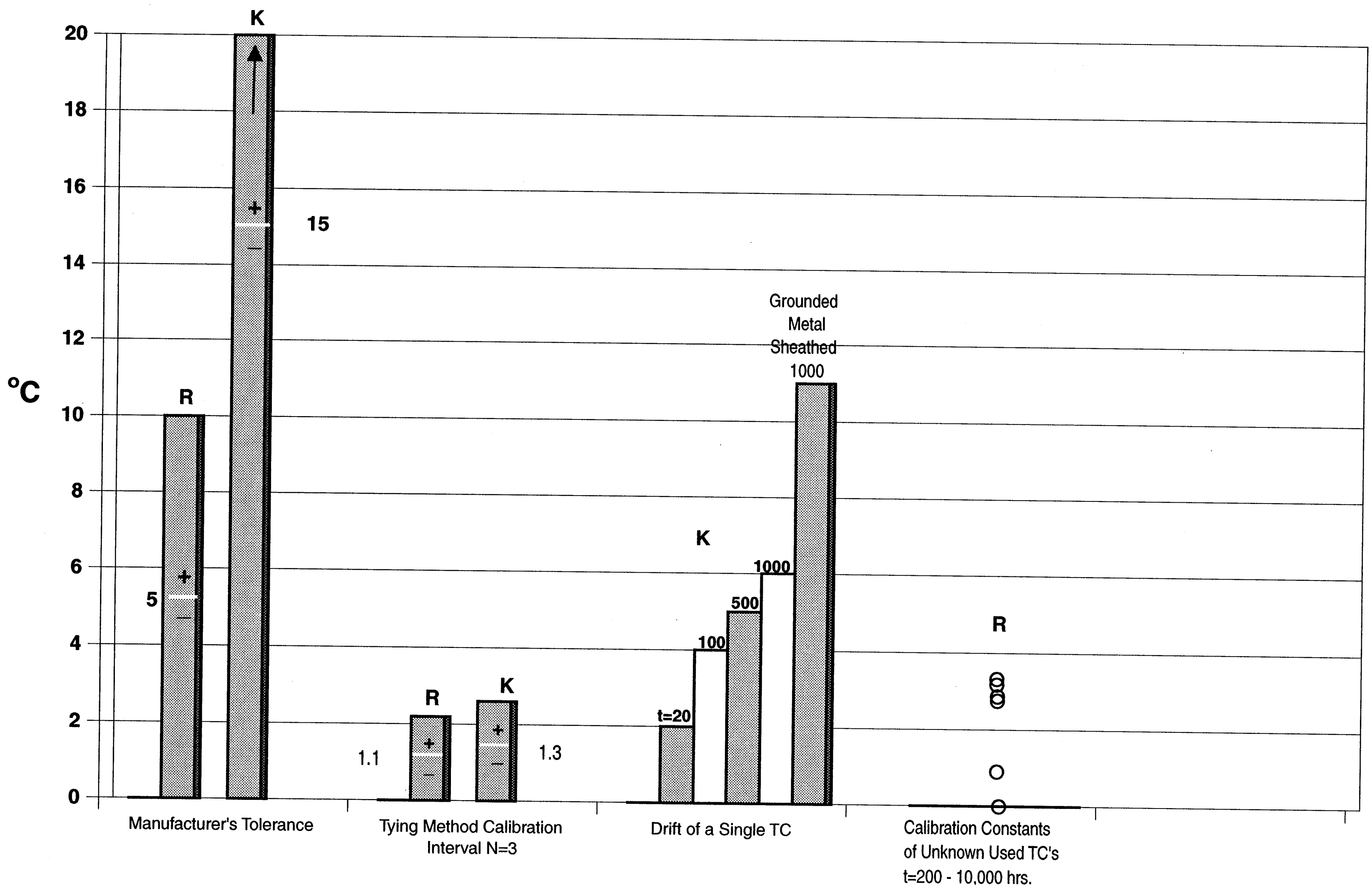


Figure A.1. Summary of thermocouple calibration experiments resulting in final testing procedures. All experiments were with sheathed type R TC's.

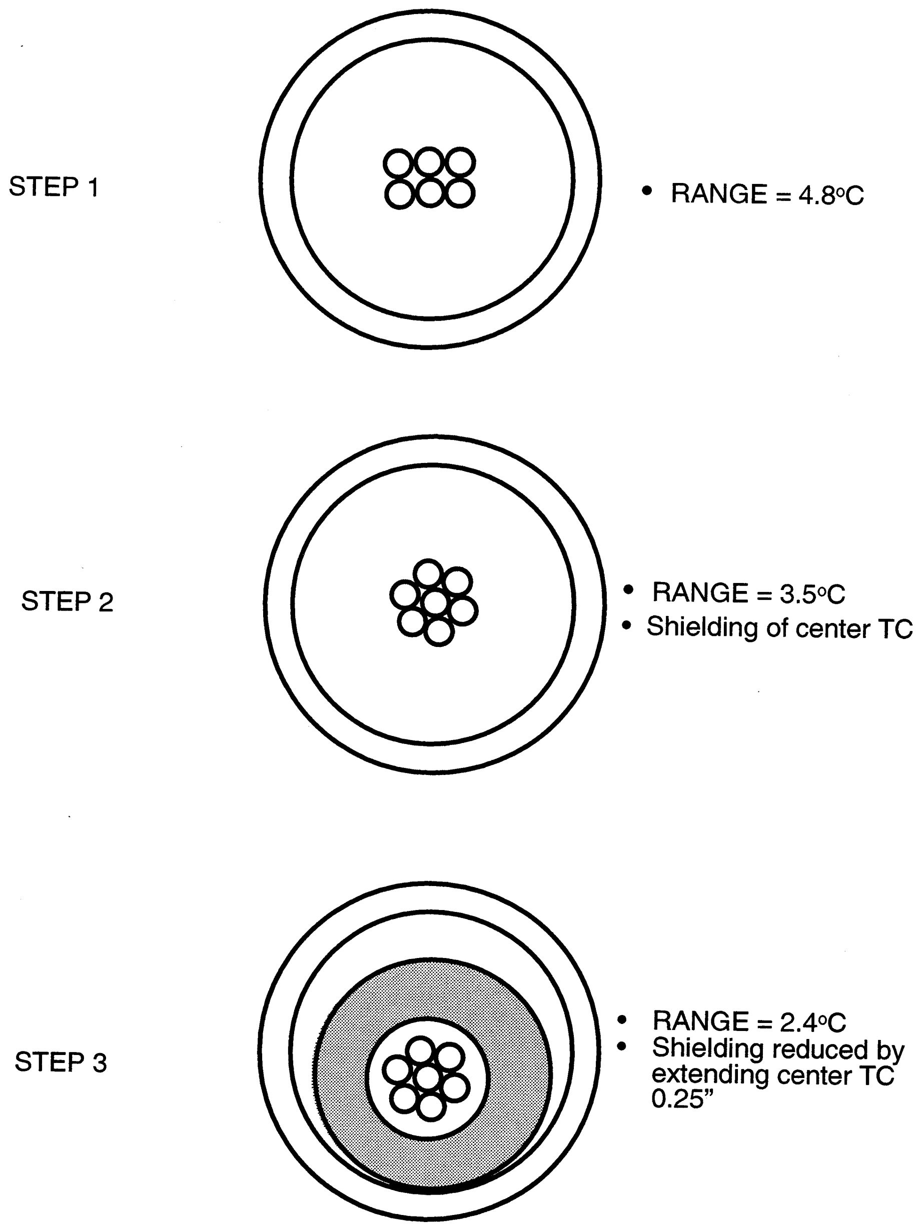
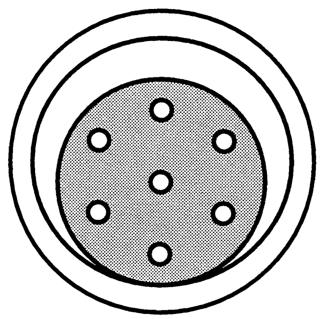


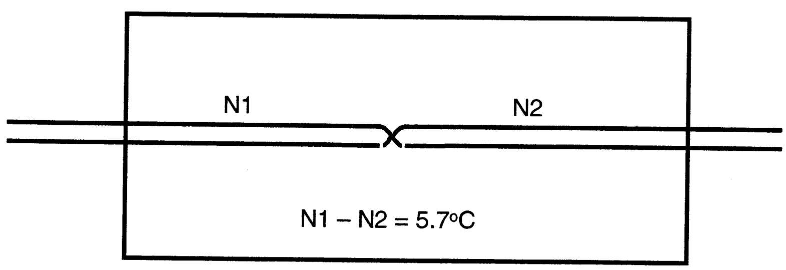
Figure A.1. continued.

STEP 4

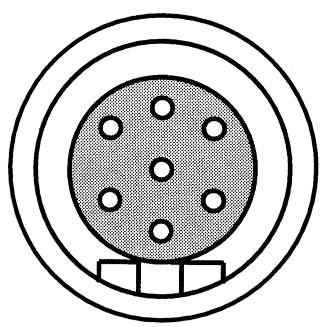


- Single run: S ~ 0.3°C
- Runs pooled S = 0.54°C
- Position offsets> 2°C

STEP 5



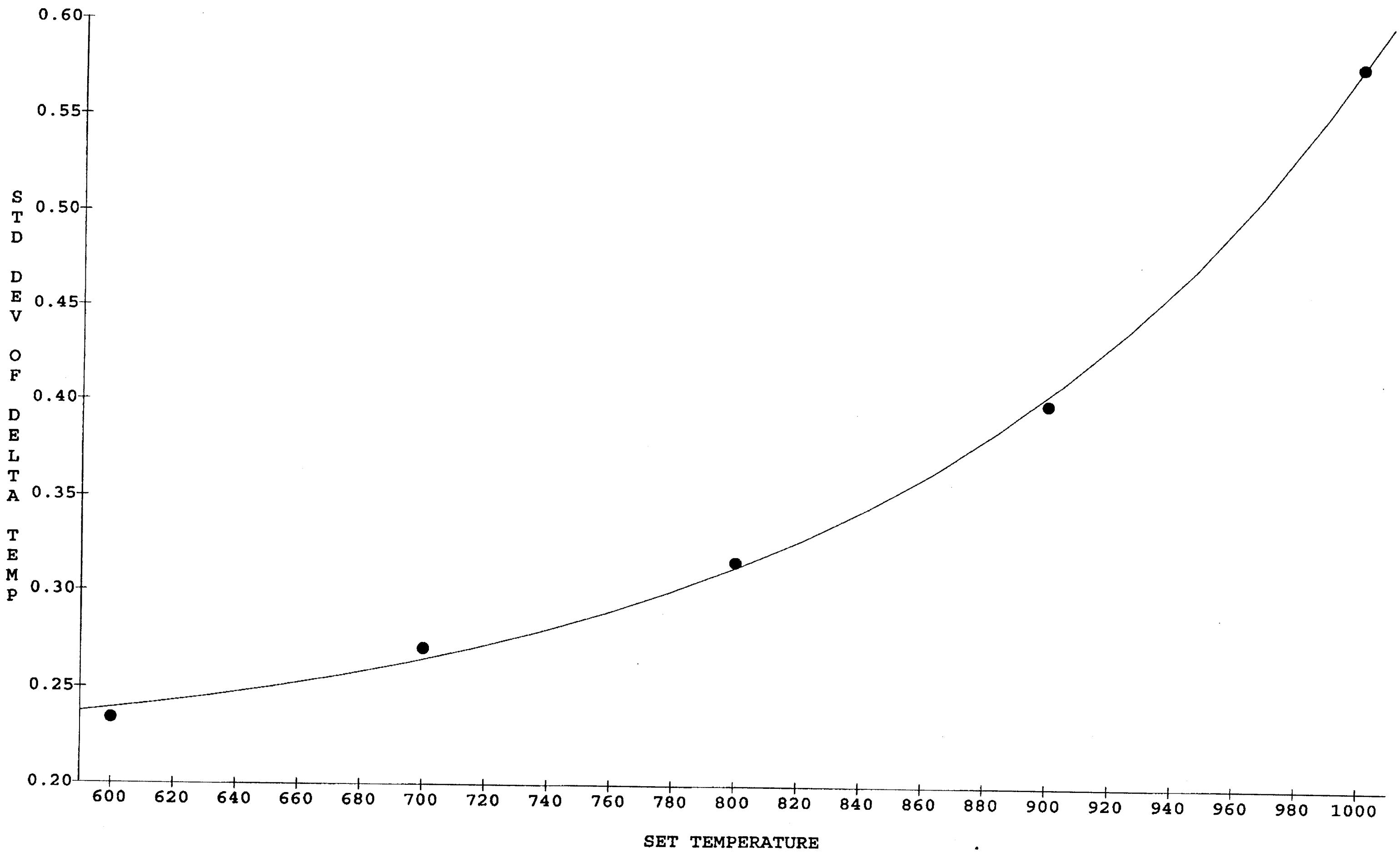
STEP 6



- Single run: S ~ 0.2°C
- Runs pooled: S = 0.41°C
- Position offsets ~ 1°C

FIGURE C1

TEST ERROR STRUCTURE FOR METAL BLOCK METHOD OF CALIBRATING UNSHEATHED THERMOCOUPLES

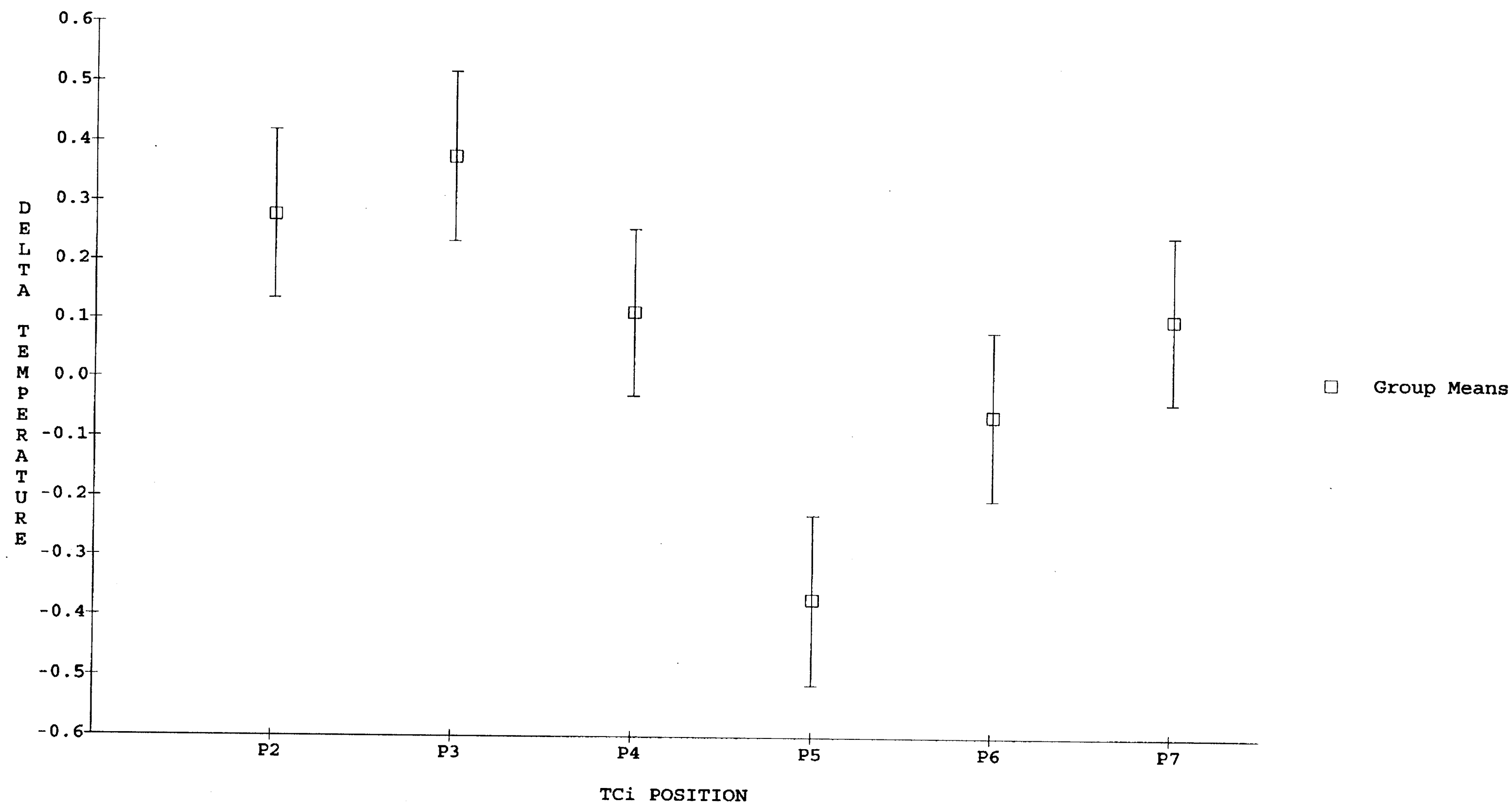


● S(DELTA T) VS. SET TEMP - RAW DATA

—— FITTED REGRESSION CURVE: Sp = 0.2106 + 0.0006315*EXP[(SET TEMP)/157.1]

FIGURE C2

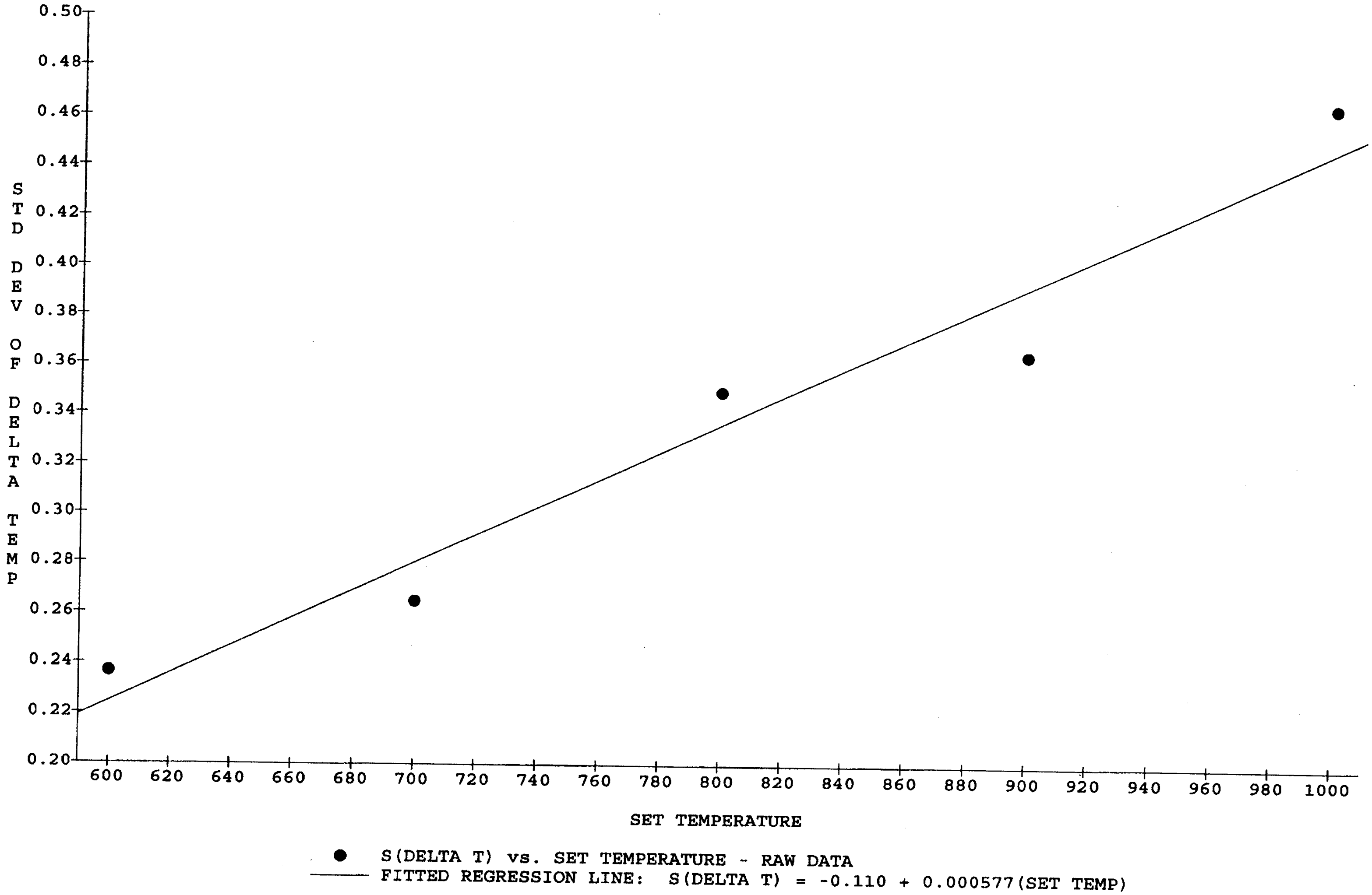
METAL BLOCK METHOD OF CALIBRATING UNSHEATHED THERMOCOUPLES
BONFERRONI MULTIPLE COMPARISON OF MEANS PROCEDURE



Error bars overlap if the difference between two means is not significant at the 5 percent level. If the error bars for the group mean overlap the dashed line, the group mean is not significantly different from the grand average. Significant differences are determined using Bonferroni simultaneous confidence intervals for all comparisons.

FIGURE D1

TEST ERROR STRUCTURE FOR TYING METHOD OF CALIBRATING UNSHEATHED THERMOCOUPLES



REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY (*Leave blank*)

2. REPORT DATE
April 2002

3. REPORT TYPE AND DATES COVERED
Technical Memorandum

4. TITLE AND SUBTITLE
Thermocouple Calibration and Accuracy in a Materials Testing Laboratory

5. FUNDING NUMBERS
WU–708–31–13–00

6. AUTHOR(S)
B.A. Lerch, M.V. Nathal, and D.J. Keller

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
National Aeronautics and Space Administration
John H. Glenn Research Center at Lewis Field
Cleveland, Ohio 44135–3191

8. PERFORMING ORGANIZATION REPORT NUMBER
E–13284

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
National Aeronautics and Space Administration
Washington, DC 20546–0001

10. SPONSORING/MONITORING AGENCY REPORT NUMBER
NASA TM—2002-211507

11. SUPPLEMENTARY NOTES
B.A. Lerch and M.V. Nathal, NASA Glenn Research Center; and D.J. Keller, Real World Quality Systems, Cleveland, Ohio. Responsible person, B.A. Lerch, organization code 5920, 216–433–5522.

12a. DISTRIBUTION/AVAILABILITY STATEMENT
Unclassified - Unlimited
Subject Categories: 35 and 04 Distribution: Nonstandard
Available electronically at http://gltrs.grc.nasa.gov/GLTRS
This publication is available from the NASA Center for AeroSpace Information, 301–621–0390.

12b. DISTRIBUTION CODE

13. ABSTRACT ***(Maximum 200 words)***

A consolidation of information has been provided that can be used to define procedures for enhancing and maintaining accuracy in temperature measurements in materials testing laboratories. These studies were restricted to type R and K thermocouples (TCs) tested in air. Thermocouple accuracies, as influenced by calibration methods, thermocouple stability, and manufacturer's tolerances were all quantified in terms of statistical confidence intervals. By calibrating specific TCs the benefits in accuracy can be as great as 6 °C or 5X better compared to relying on manufacturer's tolerances. The results emphasize strict reliance on the defined testing protocol and on the need to establish recalibration frequencies in order to maintain these levels of accuracy.

14. SUBJECT TERMS
Thermocouple; Temperature measurement; Calibrating; Accuracy; Statistics analysis

15. NUMBER OF PAGES
58

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
Unclassified	Unclassified	Unclassified	

NSN 7540-01-280-5500

Standard Form 298 (Rev. 2-89)
Prescribed by ANSI Std. Z39-18
298-102